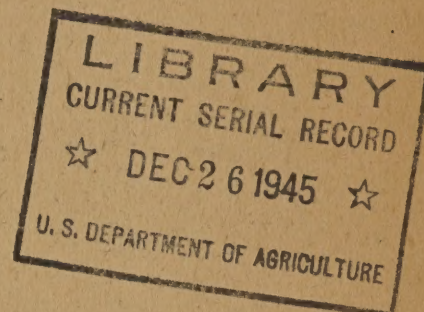


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U. S. DEPARTMENT OF AGRICULTURE  
AGRICULTURAL RESEARCH ADMINISTRATION  
BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY

O. E. May, Chief of Bureau

Report on Research Activity  
of the  
REGIONAL RESEARCH LABORATORIES  
in connection with  
the Sixth Annual Meetings  
of the



AGRICULTURAL EXPERIMENT STATIONS RELATIONS COMMITTEES

<u>Region</u>	<u>Place</u>	<u>Dates</u>
Southern	New Orleans, La.	March 27-28, 1945
Western	Albany, Calif	April 5-7, 1945
Eastern	Wyndmoor, Pa.	May 22-23, 1945
Northern	Peoria, Ill.	May 14-16, 1945

Contents

Southern Regional Research Laboratory
Western Regional Research Laboratory
Eastern Regional Research Laboratory
Northern Regional Research Laboratory

Page  
1  
15  
23  
38







# SOUTHERN REGIONAL RESEARCH LABORATORY

D. F. J. Lynch, Director

## Divisions and Division Leaders

Sym- bol	Division	Leader
A	Analytical, Physical Chemical, and Physical	T. H. Hopper
C	Cotton Chemical Finishing	J. D. Dean
F	Cotton Fiber Research	W. K. Ward, Jr.
P	Cotton Processing	R. J. Cheatham
E	Engineering and Development	E. A. Gastrock
O	Oil, Fat, and Protein	K. S. Markley
S	Sweetpotato Products	P. R. Dawson

## Commodities

- 2-3 Cotton
- 2-4 Sweetpotatoes
- 2-5 Peanuts

## Research Projects

	Page
A-4 Analytical, physical chemical, and physical investigations of cotton, sweetpotatoes, and peanuts . . . . .	2
C-1 Additive finishing of cotton textiles . . . . .	3
C-2 Modified finishing of cotton textiles . . . . .	3
C-3 Testing chemically finished textiles . . . . .	4
F-1 Relationship of chemical and physical structure of cotton fiber to technically significant properties . . . . .	4
F-2 Improvement of cotton fiber properties . . . . .	5
F-3 Colloid properties of the cotton fiber . . . . .	5
F-4 Nature and prevention of degradation of the cotton fiber . . . . .	5
F-5 Chemical derivatives of cotton fiber . . . . .	6
P-1 Development of cotton products to meet specific use requirements . . . . .	7
P-2 General relationships between fiber, yarn, and fabric properties . . . . .	7
P-3 New and improved cotton processing machinery and testing equipment . . . . .	8
P-4 Utilization of cotton and competing fibers and products . . . . .	8
E-2 Cottonseed, sweetpotato, and peanut storage investigations . . . . .	9
E-3 Cottonseed and peanut products processing investigations . . . . .	9
E-4 Sweetpotato products processing investigations . . . . .	10
O-1 Flavor and odor stability (rancidity) investigations on cottonseed and peanut oils and their derived products . . . . .	10
O-2 Oil and oilseed processing research investigations . . . . .	11
O-3 Chemistry and utilization investigations on cottonseed and peanut oils and byproducts produced during processing of these oils . . . . .	12
O-4 Chemical, technological, and utilization investigations on cottonseed and peanut meals and proteins . . . . .	12
S-1 Sweetpotato starch investigations . . . . .	13
S-2 Sweetpotato products and byproducts, other than starch . . . . .	14



Analytical, Physical Chemical, and Physical Division

RESEARCH PROJECT RRL-2-(3,4,5)-A-4. - Analytical, Physical Chemical, and Physical Investigations of Cotton, Sweetpotatoes, and Peanuts.

The work on other research projects has been assisted by providing (a) miscellaneous chemical analyses involving the application of approximately 100 techniques and increasing numbers of special research samples requiring individual attention, (b) physical chemical measurements and research services, such as viscosity, freezing point, pH, and density-composition data on mixtures of oils and solvents, and (c) specialized physical measurements such as qualitative and quantitative spectrochemical analysis, quantitative spectrophotometric analysis, refractive index, optical rotation, X-ray diffraction, and photography.

Low moisture contents and storage in sealed cans at 0°C. or below have been demonstrated to be conditions for indefinite storage or preservation without appreciable change in chemical factors of cottonseed and unshelled peanut samples reserved for experimental purposes.

An improved method has been developed for the determination of starch in sweetpotato products and other plant material. It is designed to give precise results for research purposes and may serve as a reference method by which rapid methods developed for survey and control purposes may be checked.

A systematic study has been made (a) of the variations of viscosity and density with iodine value and temperature of refined and hydrogenated peanut oils and (b) of the variations in viscosity and density with temperature of 0 to 100 percent range of mixtures of refined cottonseed, peanut, and soybean oils with Skellysolve B, acetone, and ethyl methyl ketone.

In cooperation with other Divisions (a) improved methods have been developed for the determination of alpha eleostearic acid in freshly extracted tung oil and for the estimation of soybean oil in admixture with cottonseed oil and (b) the methods of rating the water repellency of treated textile fabrics have been examined with the indication that no one method alone gives the desired information.

A number of the factors influencing the hysteresis and related elastic properties of tire cord have been studied. An apparatus has been constructed and is being used for investigating the influence of temperature and moisture on the hysteresis of different tire cords as one of the possible means of indicating the service that may be expected of the different cords.



Cotton Chemical Finishing Division

RESEARCH PROJECT RRL-2-(3)-C-1. -Additive Finishing of Cotton Textiles.

An investigation covering the formulation and application of specific compounds has been made to permit the selection of chemical treatments that afford the most effective protection to cotton textiles against the attack of microorganisms and the degrading influence of weather. The factor of penetration of individual treating agents into cotton fabrics when applied by conventional padding methods has been studied, and the improved performance of thoroughly penetrated cloth demonstrated by tests. Cotton fabrics of various types, including sandbag, camouflage and tent materials for Army use, have been treated with different preservative compounds and tested by prolonged exposure to soil contact and to weather. It is believed that certain treatments of this type can be advantageously applied to numerous fabrics in farm and in industrial use.

Selected finishing compounds, chiefly of modified cellulose and plastic types, have been formulated and applied to a standard cotton fabric with the purpose of increasing its strength and durability. Optimum conditions of application by padding methods, including temperatures and concentrations of solutions, roller pressures, etc., have been determined for individual finishing agents, and specimens of cloth finished with these compounds are being evaluated by an extended series of tests. The compounds so far examined include plain and modified starch, ethyl cellulose, alkali-soluble cellulose ether, and several resins.

RESEARCH PROJECT RRL-2-(3)-C-2. - Modified Finishing of Cotton Textiles.

Finishing treatments designed to produce permanent modification of cotton, either physically or chemically, have been employed to extend the field of usefulness of cotton textiles. Partial acetylation has been found to render yarns and fabrics highly resistant to mildew and rotting, and steps have been taken to develop a practical commercial processing method for this type of treatment. Special properties not heretofore utilized that are imparted to cotton by mercerizing with caustic soda solutions have been employed in several important textile developments. It has been found possible by this process to produce a heat-resistant cotton yarn suitable for use in tire cord, and a new semi-elastic bandage fabric made by a mercerizing treatment of ordinary surgical cotton gauze has proved of such interest to the medical profession that its commercial production appears desirable. Mercerization has also contributed to an entirely new type of unlined cotton firehose. A fundamental study of the changes effected in cotton cellulose by industrial kier-boiling and bleaching processes is being conducted with the purpose of securing important data for the cotton finishing industry.



RESEARCH PROJECT RRL-2-(3)-C-3. Testing Chemically Finished Textiles.

The possibility of developing dependable accelerated aging tests for finished cotton textiles, a subject of much scientific and industrial interest, is being extensively investigated. In this connection many actual weather exposures of plain and treated fabrics have been made and data on the resulting degradation of the cellulose collected. The destructive action on cotton of light, heat, and moisture, alone and in combinations, is being tested to secure accelerated cellulose breakdown comparable to that resulting from the action of weather. Numerous samples of cotton fabrics that have received treatments designed to afford protection against water, fire mildew, etc., are being subjected to the action of microorganisms and of weather to determine the effectiveness of the various treating agents employed. A large amount of physical testing for strength-change, abrasion resistance, water resistance, color fastness, etc. is being performed in the evaluation of textiles finished by various methods.

Cotton Fiber Research Division

RESEARCH PROJECT RRL-2-(3)-F-1. Relationship of Chemical and Physical Structure of Cotton Fiber to Technically Significant Properties.

One portion of the work now under way on this project consists of determining the structure and properties of cotton, devising suitable methods where necessary. We have been particularly interested in the microscopical structure and have developed several methods for studying this structure. A new method for the determination of refractive index is also being developed. Still another new method is nearing completion for the determination of the ratio of crystalline to amorphous cellulose in various cotton products. There is reason to believe that this ratio is very closely connected to certain of the technically important physical properties of the products.

The flex life of cotton tire cords, as determined on the testing machine developed by Dr. Karrer and Mr. Grant should be related to the other properties of the cotton fiber in these cords on one hand and to the service properties of the cords themselves on the other. This flex life appears to be an important property of the tire cord and efforts are being made to correlate it with fiber properties.

Work done under this project on the effect of swelling and stretching treatments upon the properties of cotton products has resulted in the development of a new treatment for cotton tire cord which produces a cord of improved properties. Evaluation of this cord in tires is now in progress by this Division and Cotton Processing Division in cooperation with the Goodyear Tire and Rubber Company.



RESEARCH PROJECT RRL-2-(3)-F-2. - Improvement of Cotton Fiber Properties.

The Cotton Fiber Research Division and the Cotton Chemical Finishing Division have been cooperating in developing new methods for improving the flameproof and fireproof properties of cotton. In the Cotton Chemical Finishing Division this work is being done under RRL-2(3)-C-1. Several novel treatments have been developed. Some of these are not suitable for use in clothing fabric, but might be applied to cotton insulation. Others may be used for uniforms and similar fabrics. The best of these retains its flameproofing effect after twelve standard launderings, but is not perfect as far as afterglow is concerned. A simple, practical method for regenerating flameproofing qualities of certain treatments removed by laundering has also been developed. Efforts to improve these methods are still in progress.

RESEARCH PROJECT RRL-2-(3)-F-3. - Colloid Properties of the Cotton Fiber.

Research under this project follows two lines. The major emphasis is being given to improving the adhesion of cotton tire cord to rubber and to synthetic rubber. A method for the measurement of this adhesion at various temperatures has been developed and a mass of data has been compiled on the adhesion of cotton and competitive tire cords. The effect of various commercial adhesives on adhesion has been determined and some new adhesives have been developed in the course of the work. Future work along this line is expected to emphasize the development of such adhesives.

Work has also been in progress on improving the technique of measuring the viscosity of cotton cellulose in various solvents, especially in cuprammonium hydroxide solution. The influence of light, heat, and chemical degradation on the viscosity is being investigated.

RESEARCH PROJECT RRL-2-(3)-F-4. - Nature and Prevention of Degradation of the Cotton Fiber.

Four types of degradation are being currently studied: thermal degradation in tire cord, microbiological degradation in fishing gear, hydrolytic degradation in fertilizer bag cord, and degradation by anhydrous acids. The last of these is an investigation of methods for following the theoretical course of such degradation reactions, while the other three deal with practical problems which are of importance to the industry and especially just now to our war efforts.

A large amount of data has been collected on the effect of heat upon cotton tire cord in view of the frequent claim that rayon possesses superior heat-resistance to cotton. The effect of elevated temperatures at various humidities upon the breaking strength of cotton tire cord is of especial interest and accurate determinations of this strength under various conditions are under way. The development of a tire cord which retains high tensile strength at elevated temperatures is also being investigated.



RESEARCH PROJECT RRL-2-(3)-F-4 (Continued)

In cooperation with the Fish and Wildlife Service of the Department of the Interior, a series of experiments in various fishing waters of this country is under way comparing a series of commercial preservatives with a furfuryl alcohol resin treatment developed at this Laboratory. At present cords are being tested in Bayou St. John, New Orleans, Louisiana, at Duxbury Harbor, Massachusetts, and at Pensacola, Florida. Other tests in the Mississippi River are being planned.

One of the problems of the Office of Production Research and Development, has been the failure of bags used in shipping Lend Lease triple superphosphate, due to acid hydrolysis of the cotton threads used in these bags. The Southern Regional Laboratory was one of the groups assigned this problem. The method developed here for the prevention of this degradation consists of treatments with various humectant agents, the best of which appears to be triethanolamine. Triethanolamine furnishes protection to the cotton thread if care is taken to avoid oxidation during application and drying.

RESEARCH PROJECT RRL-2-(3)-F-5. - Chemical Derivatives of Cotton Fiber.

Three types of work are being carried on under this project. One of these consists of the chemical modification of the cotton in tire cord in order to improve its properties; in other words the formation of derivatives in situ in the cord either on the surface or throughout the mass of the fiber. Impregnations with various chemicals are also being studied under this line project.

A second type of work on chemical derivatives is that dealing with cellulose nitrate. A method has been developed for stabilizing nitro-cotton which effects the saving of some 26 hours in the boiling time required for the preparation of smokeless powder. This method has been used successfully on a 7200 pound run at the Naval Powder Factory, Indian Head, Maryland. The efficacy of this treatment for lacquer and plastic nitrocellulose is now being studied.

The Cotton Fiber Research Division is cooperating with the Cotton Chemical Finishing Division under Project RRL-2(3)-C-2 on the acetylation of cotton products to give either partial or complete conversion to cellulose acetate. Along with this work we have been cooperating with the Division of Cellulose Chemistry of the American Chemical Society in the development of standard methods for the determination of acetyl and formyl contents in cotton and other cellulosic materials.



Cotton Processing Division

RESEARCH PROJECT RRL-2-(3)-P-1. - Development of Cotton Products to Meet Specific Use Requirements.

Research under this project is at present being devoted almost exclusively to the development of improved types of cotton tire cord through the experimental selection of commercially available varieties of cotton best suited for this use. Experimental selection is being accomplished by means of laboratory and service tests on cord made from a number of varieties selected for this work through the cooperation of the Bureau of Plant Industry, Soils, and Agricultural Engineering. Three varieties of cotton (Wilds 13, Stoneville 2-B and SxP) have been processed into cord and made into tires and tested by the Army and the War Production Board. To date, six service tests have been run, three by the Army and three by the War Production Board. In the last three tests, Wilds cord was the only experimental cord used, this variety having been selected as outstanding on the basis of results obtained from the first three service tests. All three varieties, however, gave definitely superior results to those obtained with regular commercial cords used as controls.

Cord is now being manufactured according to commercial practice from Wilds for use in a large-scale service test by the Goodyear Tire and Rubber Company. This test will include, at the same time, Wilds cord given a special stretching process developed by the Cotton Fiber Research Division (see RRL-2(3)-F-1) and some of Goodyear's regular commercial cord.

As an aid in the development of cotton products to meet specific use requirements, special studies are being made to determine (1) the relationship of cord construction to the flex-life of tire cord, and (2) the effect of heat upon the physical properties of tire cord made from cotton and competing materials.

RESEARCH PROJECT RRL-2-(3)-P-2. - General Relationships between Fiber, Yarn, and Fabric Properties.

Current work under this project has as its objective the determination of optimum twist combinations in the single, ply, and cable of cotton tire cord. One variety cottons ranging from 1" to 1-1/2" in length are being used in the experiments. For screening tests the optimum is being judged on the basis of laboratory tests for tensile strength, elongation, adhesion, flex-life, uniformity, etc.

Studies are also being made to experimentally determine what roll settings and drafts should be used for different staple lengths of known varieties of cotton to obtain optimum qualities of yarns. In these tests the strength, uniformity, and appearance of the single yarns is being used as criteria. The fiber strength and other measurable physical properties of each lot of cotton, used in these experiments, is being assembled with a view of correlating these fiber properties with the physical properties of the yarns and the percentages of waste removed during processing.



RESEARCH PROJECT RRL-2-(3)-P-2. (Continued)

Studies to determine the general relationships between fabric structure and fabric properties will be undertaken as time permits.

RESEARCH PROJECT RRL-2-(3)-P-3. - New and Improved Cotton Processing Machinery and Testing Equipment.

There are two distinct phases to the work undertaken under this project. The first consists of studies of methods and equipment now employed for processing and testing cotton products to secure the necessary information for the development of more efficient equipment. The second phase involves the design and experimental development of the equipment.

One major line of research under this project is the development of the high-capacity, efficient machine for cutting lint cotton to lengths suitable for purification and manufacture into smokeless powder on existing commercial equipment now being used to process linters. Cutting machines commercially available do not have the required capacity. Following the designing, development and successful operation of a pilot size cutter, a commercial cutter designed by and built under the supervision of engineers of the Cotton Processing Division has now been installed at the lint purification plant of the Buckeye Cotton Oil Company for large-scale tests. Preliminary runs have shown that the machine has a capacity about five times that of available commercial cutters, but that minor changes are necessary to permit satisfactory continuous operation. At the request of the War Production Board, these changes are being made with the greatest possible speed.

Other research includes the development of a machine for slashing cotton textile warp yarns, which will eliminate the harsh treatments given warp yarns in this process on present machines; the development of an electronic method for rapidly drying cotton textiles under closely controlled conditions; and the design and development of a special device to stretch and dry tire cord at high speeds.

RESEARCH PROJECT RRL-2-(3)-P-4. - Utilization of Cotton and Competing Fibers and Products.

Research under this project consists of securing, analyzing, and interpreting economic and technical data needed in selecting, guiding, and appraising technical and scientific research projects. This work at present falls into two main categories: (1) the use of cotton and competitive products in tire cord, and (2) miscellaneous short-time surveys and appraisals on the utilization of cotton and competing fibers in other products.

The work on tire cord consists of obtaining and keeping up-to-date such information as production capacities for cotton and rayon tire cord, processing costs and prices of different qualities of cord, physical properties of cord and trends in the use of cord of different constructions and properties, etc. Technical data is also obtained on the



RESEARCH PROJECT RRL-2-(3)-P-4. (Continued)

performance of cords of different types in actual service through contact with tire manufacturers and through observation of service tests conducted on tires made from Southern Regional Laboratory experimental cords by the Army and War Production Board. A critical study is now being made of the results obtained from standard laboratory testing machines in order that they may be properly interpreted in terms of fundamental physical properties of the materials tested. This information is useful, not only in interpreting and appraising the relative worth of various types of cords for use in tires, but also in providing information which will be helpful in the modification of present standard laboratory testing machines and in the design of new types of machines.

Owing to the constantly changing nature of short-time surveys and appraisals, a description of current work by name would have little meaning. Examples can be given, however, which will be indicative of the types of research which this work involves: A survey has recently been completed on the development and use of rayon and other synthetic fibers, and a survey and appraisal is now being made of the mechanical and chemical properties, production and price of various new types of synthetic fibers and the products made therefrom. An extensive file, which is kept systematically up to date, provides much of the information needed in supplying other sections of this Division, and other Divisions of the Laboratory, with the economic and technical data needed in connection with their research projects. Other information is obtained from field surveys and through correspondence.

Engineering and Development Division

RESEARCH PROJECT RRL-2-(3,4,5)-E-2. - Cottonseed, Sweetpotato, and Peanut Storage Investigations.

Facilities are available for storage of cottonseed, peanuts, and sweetpotatoes under conditions industrially used. Storage experiments on pilot plant scale have been made on cottonseed.

RESEARCH PROJECT RRL-2-(3,5)-E-3. - Cottonseed and Peanut Products Processing Investigations.

Pilot plant processing investigations have been carried out on cottonseed and peanut preparation, including cleaning, delinting, hulling, and flaking. Processes for the production of cottonseed and peanut oils, proteins and their derived products from residual meal, have been investigated. These processes include hydraulic pressing, expeller pressing, and solvent extraction of the cottonseed and peanut flakes; solvent crystallization; separation, and purification of proteins derived from hydraulic pressed meal or solvent extracted meal to permit the utilization of proteins in collaborative work on adhesives and fibers.



RESEARCH PROJECT RRL-2-(3,5)-E-3. (Continued)

Pilot plant equipment for processing operations now being installed for the production of cottonseed and peanut oils, includes continuous solvent extraction, hydrogenation, refining, bleaching, deodorization, facilities for fat splitting under high temperature and pressure conditions, special oil and glyceride reactions at temperatures up to 600°F. and pressures from vacuum to about 30 pounds.

RESEARCH PROJECT RRL-2-(4)-E-4. - Sweetpotato Products Processing Investigations.

The sweetpotato starch pilot plant has been altered and improved in layout and equipment to permit balanced operation as a complete unit in production of high-grade starch by several methods of purification. At the same time, provision has been made for experimental study of the following unit operations: Washing, grinding, screen separation, purification by centrifugal hydro-separation, settling or tabling, bleaching, and modification of starch, dewatering and drying of starch and pulp, and byproduct recovery.

Several successful runs have been made with effort concentrated on purification of starch with the semi-plant-size continuous high-speed centrifugal simulating in performance the large commercial units being installed in the new sweetpotato starch factory approaching completion by the U. S. Sugar Corporation at Clewiston, Florida. These facilities and the findings of the investigations will materially aid effective technical assistance to the new starch enterprise in development of procedures and techniques for operation and control of its large industrial installation at maximum efficiency for production of high quality starch.

Oil, Fat, and Protein Division

RESEARCH PROJECT RRL-2-(3,5)-O-1. Flavor and Odor Stability (Rancidity) Investigations on Cottonseed and Peanut Oils and their Derived Products.

Work on this project may be divided into two phases, one of which involves the stability of the oil in the unprocessed or whole seed, and the other involving the oil after removal from the seed.

Work on the pre-treatment of cottonseed prior to storage for the purposes of preventing or reducing losses of neutral oil during storage, has been extended from pilot plant to semi-commercial scale tests. Four oil mills located in the states of Texas, Louisiana, Mississippi, and Georgia, have participated in this work. A total of 325 tons of seed were used in these storage tests, divided as follows: Three 30-ton lots treated with ammonia, four 30-ton lots treated with a synthetic detergent, and four controls totaling 115 tons. Owing to various difficulties, especially with regard to available labor and material at the oil mills, the experiments were started too late in the season



RESEARCH PROJECT RRL-2-(3,5)-0-1. (Continued)

to obtain the maximum benefit from the treatment of the seed, as well as the maximum desired amount of information concerning the process, as would have been possible if the experiments could have been started earlier and with wetter seed. However, enough information is now available, and more will be available by the time the seed is crushed, to determine the relative effects of the various treatments and the general requirements for further mill scale tests under more favorable conditions.

The pilot plant work conducted during the current year was based on the results of several years of laboratory investigations on the respiration of cottonseed and the natural and artificial factors which affect this process.

Considerable work has been conducted on the stability of both cottonseed and peanut oils and on products, such as peanut butter, which contain these oils. This work has included separation of the antioxidant concentrates from peanut and cottonseed oils by molecular distillation, steam distillation, low temperature solvent fractionation, and chromatography; the effect of natural and synthetic antioxidants on the stability of these oils; development of methods of estimating the presence of tocopherols in natural fats; identification of the product produced from gamma-tocopherol during oxidation of cottonseed and peanut oils; and the effect of metallic salts on the stability of hydrogenated cottonseed oils used as a substitute for palm oil in the tin-plate industry.

RESEARCH PROJECT RRL-2-(3,5)-0-2. - Oil and Oilseed Processing Research Investigations.

Work on this project has included a variety of investigations relating to the development of new products from cottonseed and peanut oils, and of new or improved processes of producing these products, as well as the development of methods for, and the measurement of, various physical properties of natural and modified fats which are of especial application and significance in the development of modified fats and oils.

The various investigations stem from a process of low temperature solvent crystallization which was developed for the purpose of winterizing cottonseed and peanut oils and for the production of modified cottonseed and peanut oils for specialty uses such as textile lubricants and assistants, filling leather, hard butters, etc. The laboratory work has been extended to the development of a pilot plant unit for producing these products by a continuous process at the rate of about 200 pounds per hour. The development, first, of laboratory scale equipment, and, later, of pilot plant equipment to accomplish these objectives was based on an extensive investigation of the thermal, dilatometric, and plastic properties of natural and hardened oils and on the chemical reactivity of fats and fatty acids under various conditions of selective hydrogenation, catalytic esterification, etc.



RESEARCH PROJECT RRL-2-(3,5)-0-3. - Chemistry and Utilization Investigations on Cottonseed and Peanut Oils and Byproducts Produced During Processing of These Oils.

Little work has been done on this project owing to concentration on war emergency problems for the U. S. Quartermaster Corps, Board of Economic Warfare, and other governmental agencies, as well as on cooperative work with several of the State Experiment Stations. Miscellaneous work of this character has included analyses of babassu nuts and oils, Coyol palm nuts and oil, rice bran oil, chicken fat produced as a byproduct of chicken packed for the U. S. Army, okra seed and oil, gutcha nut oil, Mexican castor beans, and Garcia Mayana seeds from the Puerto Rico Agricultural Experiment Station.

More specifically related to the project has been the development of a method of detecting the presence of soybean oil in cottonseed and peanut oils, a modified thiocyanogen reagent and method for estimating the extent of the unsaturation in the above-mentioned oils, and an improved method of removing the solvent from solvent-extracted oils prior to determining the content of free fatty acids.

RESEARCH PROJECT RRL-2-(3,5)-0-4. - Chemical, Technological, and Utilization Investigations on Cottonseed and Peanut Meals and Proteins.

Work on this project has covered three distinct phases, namely fundamental work on cottonseed and peanut proteins and meals, development of protein adhesives, and development of protein fibers. The fundamental work on cottonseed and peanut meals has been concerned with the nature of the pigments which are present in the intact seed, in the processed meal, and in the isolated protein, and with their effects on the color of finished products produced from these commodities. Three previously unidentified pigments have been isolated from cottonseed in addition to the well-known gossypol. These pigments which are colored orange, purple, and blue, have been designated gossyfulvin, gossypurpurin, and gossycaerulin, respectively. Work completed to date indicates that they are structurally related to gossypol and are in part, at least, produced from gossypol during processing of the seed. Two of them also occur naturally in the seed prior to processing but may be increased in amount by processing. New methods of estimating gossypol and of determining the other pigments have been developed.

Other fundamental work on cottonseed and peanut proteins has been directed toward the isolation of pure protein fractions and methods of determining certain of their amino acid constituents; and the development of an in vitro digestibility test for protein meals, investigation of the solubility and peptization phenomena of cottonseed and protein meals.

Fractical work on the utilization of peanut proteins has led to the development of adhesives which in laboratory and practical tests have been shown to possess properties adapting them to use in the manufacture of gummed tape, postage stamps, envelope glues, etc., and as flexible glues for use in the manufacture of setup paper boxes, bookbinding, etc., also as plywood glues comparable with casein and soybean glues.



RESEARCH PROJECT RRL-2-(3,5)-0-4. (Continued)

Work on the development of peanut protein fiber has been carried to the stage of laboratory production of fibers equal in quality to present casein and soybean fibers.

Sweetpotato Products Division

RESEARCH PROJECT RRL-2-(4)-S-1. - Sweetpotato Starch Investigations.

Major efforts have continued to be directed toward improving the efficiency and the control of unit operations and processes in extraction, purification, and finishing of sweetpotato starch and toward control of the uniformity and quality of the product to preserve its desirable and superior characteristics and to adapt it most effectively to the requirements of specific uses.

More complete and reliable data have been obtained on the distribution of the starch, water, pulp, and soluble components in counter-current screening of starch from pulp, and in starch concentration and purification by settling or by high speed centrifugation. Additional experimental and empirical plant information have been acquired to guide improved control of acid modification of starch for products of different degrees of paste viscosity, to guide optimum control of the modifying influences in bleaching and drying of starch. The feasibility of reliable electrometric control of pH and lime water concentration in processing has been demonstrated. Investigations have been intensified on current practices and requirements in such major and specialized applications of starch as warp sizing to guide appropriate control of starch quality in the finishing and blending processes. Further improvements in analytical and other laboratory procedures have facilitated reliable factory control from raw material input to finished product. During the 1944 operations in the Laurel plant new and up-to-date data were obtained on over-all and unit manufacturing costs.

Effective application of the findings of these investigations has been made in continued technical assistance to the new sweetpotato starch enterprise of the U. S. Sugar Corporation at Clewiston, Florida, with whom there is a cooperative agreement. With the plant rapidly approaching completion, and with test runs scheduled for late March or April, accumulated findings and data from work in progress will find even more important application in guiding adjustments and control of equipment and processes for efficient operation, with output of uniform and high quality products. The expedition with which this large modern plant attains full-scale production, marking the establishment of the sweetpotato starch industry, depends on the facility with which the sweetpotato starch manufacturing process as developed to small commercial scale in the experimental plant at Laurel is translated to the equipment and volume of the new plant.



RESEARCH PROJECT RRL-2-(4)-S-2. - Sweetpotato Products and Byproducts,  
Other than Starch.

Continued progress has been made in development of practical processes for increasing the byproduct credit in processing of sweetpotatoes for starch and other products. Continuous coagulation and concentration of the crude protein now wasted in starch plant fruit water appear particularly successful when applied to the effluent from centrifugal concentration of starch milk; and a simple and low-cost process for collecting the crude protein on spent pulp thus raising the protein content of the byproduct feed appears feasible. Some 55 percent of the nitrogen in the crude protein has been quantitatively identified in the form of 14 amino acids, most of them essential. The crude concentrate of protein and other material where yellow or orange-fleshed sweetpotatoes are processed is also a high carotene concentrate.

Pending delivery of equipment for large-scale operations, investigations have been concentrated on further raising the yield of feed yeast, produced from the sugars in starch factory waste water, from the present maximum of around 44% of the sugar content; refinements have been made in the technique of propagation contemplating large-scale application; and the feasibility of continuous propagation has been demonstrated, yielding much more concentrated yeast slurries.

Improvements have been made in the processes for removal of residual starch from sweetpotato pulp to permit extraction of high grade pectin.

Recovery of sweetpotato protein and production of feed yeast as byproducts of sweetpotato starch manufacture offer particular promise for application in a continuous process as contemplated in the starch enterprise at Clewiston, Florida, would materially increase the value of the byproduct feed, and would reduce the load on the waste disposal plant.

Technical assistance has been given the sweetpotato dehydration enterprise of Sweet Potato Growers, Inc., at Laurel, Mississippi, in improvement and maintenance of equipment and in control of quality in processing. With high quality sweetpotatoes to start with, considerable variation in the time and conditions of storage do not seriously affect the quality of the dehydrated product.

Anticipating the importance of livestock in post-war adjustments of Southern agriculture and the need for extended outlets for crops other than cotton, work is being undertaken to obtain more satisfactory information than is now available on the most economical equipment and process for dehydration of sweetpotatoes for stock feed. To contribute to the necessary lowering of costs of sweetpotato production if starch manufacture or feed production are to be feasible under most conditions, investigations are being undertaken on processes for economical dehydration of sweetpotato vines to facilitate credit from the potentially valuable byproduct of sweetpotato production.



WESTERN REGIONAL RESEARCH LABORATORY

T. L. Swenson, Director

Divisions and Division Leaders

Sym- bol	Division	Leader
B	Biochemical	H. D. Lightbody
CB	Commodity Byproducts	L. B. Howard
CP	Commodity Processing	R. P. Straka (actg.)
ED	Engineering and Development	W. B. Van Arsdell
PA	Physicochemical and Analytical	C. H. Kunsman
P	Protein	G. H. Brother (actg.)

Commodities

- 3-3 Apples
- 3-4 Alfalfa
- 3-5 Fruits
- 3-6 White potatoes
- 3-7 Poultry products
- 3-8 Vegetables
- 3-9 Wheat
- 3-10 Western hemlock bark

Research Projects

Page

B-6	The laboratory preparation of enzymes from wastes, culls, and surpluses, and the use of enzymes in both new and developed food and industrial processes. . . . .	16
B-7	Utilization of fruit, vegetable, and wheat surpluses, culls, wastes, and processing residues by microbiological processes. . . . .	17
B-8	Use of agricultural commodities as sources of therapeutic agents and nutritional supplements. . . . .	17
B-9	The control of quality-deteriorating biochemical agents and reactions, before and during processing and during storage of the finished products. . . . .	17
CB-6	Preparation, properties, and utilization of glyceride oils and fats derived from fruits, vegetables, and poultry products. . . . .	17
CB-7	Preparation, determination of constitution and properties, and utilization of pectic substances. . . . .	18
CB-8	Miscellaneous byproduct recovery and utilization . . . . .	18
CB-9	Dehydration processing and evaluation investigations . . . . .	18
CP-5	Investigations of the preservation at freezing temperatures of apples, fruits, poultry products and vegetables with reference to the influence of variety, type or breed, maturity or age, harvesting, grading, handling, prefreezing treatment, packaging, freezing, storage, and transport. . . . .	19
CP-6	Investigations of the microbiology and histology of apples, fruits, poultry products, and vegetables preserved by freezing at low temperatures . . . . .	19



	Page
CP-7 Investigations of the utilization of fruits, poultry products, and vegetables preserved by freezing at low temperatures . . . . .	19
ED-6 Engineering and economic investigations in the industrial processing of Western agricultural commodities for food.	20
ED-7 Engineering and economic investigations in the recovery of byproducts from the industrial processing of western agricultural commodities . . . . .	20
ED-8 Engineering and economic phases of the development of high-grade alfalfa feeds . . . . .	20
PA-4 Macrochemical and microchemical analyses of the agricultural commodities and the products derived therefrom . .	21
PA-5 X-ray, microscopic, crystallographic, spectroscopic, color, electronic and other physical methods of testing and analyzing agricultural materials and the products derived therefrom. . . . .	21
PA-6 Colloidal, chromatographic, spectrophotometric, fluorometric, photochemical, heat transfer, reaction kinetics, and other physical chemical investigations pertaining to the agricultural commodities and the products derived therefrom. . . . .	21
P-3 Chemical composition, reactions, and reactivity of assigned commodity proteins. . . . .	22
P-4 Investigation of structure and structural and physical chemical relationships in alfalfa, poultry, and wheat proteins . . . . .	22
P-5 Industrial application and utilization of assigned commodity proteins. . . . .	22

### Biochemical Division

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-B-6. - The Laboratory Preparation of Enzymes from Wastes, Culls, and Surpluses, and the Use of Enzymes in both New and Developed Food and Industrial Processes.

Enzyme preparations are used in the textile, paper, adhesive, pectin, candy, laundry, leather, cheese, silk, and photography industries and as laboratory preparations or analytical reagents and in therapeutics. Vegetable and fruit wastes are being investigated as possible sources of commercially useful enzymes, both by isolation of the enzyme in the wastes and by use of the wastes to grow microorganisms that produce enzymes.

Proteinase has been produced in high yield by culturing Bacillus subtilis on waste asparagus-butt juice. Citrus peel residue is being investigated as a practical source of a pectin-de-esterifying enzyme.



RESEARCH PROJECT RRL-3-(3,5,6,8,9)-B-7. - Utilization of Fruit, Vegetable, and Wheat Surpluses, Culls, Wastes, and Processing Residues by Microbiological Processes.

Canning plant wastes containing large quantities of sugars have been shown to be possibly useful for feed yeast production. The juices of some vegetable wastes have been shown to be excellent culture media for some micro-organisms which produce antibiotic agents used in control of infections and probably useful for controlling some plant diseases. The researches in progress include investigation of conditions required for yeast production, preparation of the yeast for marketing, preparation of marketable juice concentrates, and extension of knowledge concerning usefulness of juices as microbiological culture media.

RESEARCH PROJECT RRL-3-(3,4,5,7,8,9)-B-8. - Use of Agricultural Commodities as Sources of Therapeutic Agents and Nutritional Supplements.

The presence of biologically active compounds of potential therapeutic usefulness has been demonstrated in agricultural commodities of both plant and animal origin. Methods are being investigated for the isolation of a germicide (lysozyme) from waste egg white and vitamin C from the hulls of Persian (English) walnuts. Other commodities will be examined as possible sources of products useful in medicine.

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-B-9. - The Control of Quality-Deteriorating Biochemical Agents and Reactions, Before and During Processing and During Storage of the Finished Products.

Investigations directed toward prevention of such deterioration in dried eggs are concerned with: (a) the locale (yolk, white, lipid, etc.) of the deteriorative changes; (b) the nature of the reactions involved (whether they involve oxidation, aldehyde-amine, hydrolysis, enzymes, lipids, or some other reaction); and (c) factors such as oxygen, moisture and pH that are known to influence deterioration.

Commodity Byproducts Division

RESEARCH PROJECT RRL-3-(5,7,8)-CB-6. - Preparation, Properties, and Utilization of Glyceride Oils and Fats Derived from Fruits, Vegetables, and Poultry Products.

Glyceride oils and fats are widely distributed in agricultural commodities. Frequently they occur in seeds or other tissues that are of little or no value in the normal utilization of the commodity. Examination of the physical and chemical properties and potential uses of the oils in Valencia orange seeds, grape and raisin seeds, apricot kernels, and avocado has been undertaken. Since domestic oils are relatively rich in saturated fatty acids, such as palmitic and stearic, the utility of which is limited, studies are in progress on modification of these saturated fatty acids, so that they can be used as



RESEARCH PROJECT RRL-3-(5,7,8)-CB-6. (Continued)

substitutes for imported oils or as more useful raw materials in industry. Current studies include chemical modification of palmitic acid to free-lathering soap acids, as potential replacements for imported coconut and palm oils. Fatty-acid derivatives useful as plasticizers are also being investigated.

RESEARCH PROJECT RRL-3-(3,5,8)-CB-7. - Preparation, Determination of Constitution and Properties, and Utilization of Pectic Substances.

Pectin is widely distributed in commodities but is commercially derived only from citrus and apples. Current production of some six million pounds annually is about double the normal pre-war production but represents only a small percentage of the pectin readily available from the processing of wastes and culls. Cheaper production and the development of derivatives with special characteristics would be of value to the expanded industry and to agriculture. More simple and economical methods of recovery of high-grade pectins from both apple and citrus materials are being studied. Chemical modification of pectin, such as partial demethoxylation, is being studied to develop products with novel and interesting uses, both in the food-processing and non-food industries. Current investigations include development of derivatives of potential value to the canning, preserving, beverage, dessert, dairy products and other food industries and possibly in the production of special fibers, films and adhesives.

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-CB-8. - Miscellaneous By-product Recovery and Utilization.

Large quantities of culls, thinnings, and processing residues are potential sources of materials such as fibers, fillers for plastic compositions or structural boards, fruit acids, aldehydes, terpenes, and similar substances. Methods are being developed for the recovery of tartaric and malic acids from pomace and still wastes in wineries. Potential sources of fruit acids in other commodities will be examined and methods for their practical recovery will be studied. Fruit and vegetable residues from processing operations will be examined as sources of volatile flavorful constituents, and uses will be sought for nut and seed-pit shells and hulls.

RESEARCH PROJECT RRL-3-(3,5,6,8)-CB-9. - Dehydration Processing and Evaluation Investigations.

Preservation of fruits and vegetables by procedures that conserve container materials and shipping and storage space has been important in wartime. Dehydration offers maximum advantages in meeting these conditions, but lack of an established industry and adequate "know how" has created problems and emphasized the need for continued research on this method of food preservation. Investigations are in progress on the effect of raw-material factors, preparation and pre-drying methods, and dehydration conditions on the ease of rehydration, nutritive value, and organoleptic characteristics of the processed materials. Factors responsible



RESEARCH PROJECT RRL-3-(3,5,6,8)-CB-9. (Continued)

for losses of nutritive and edible quality during storage of the dry products are being studied and methods of avoiding such deterioration developed.

Commodity Processing Division

RESEARCH PROJECT RRL-3-(3,5,7,8)-CP-5. - Investigations of the Preservation at Freezing Temperatures of Apples, Fruits, Poultry Products and Vegetables with Reference to the Influence of Variety, Type or Breed, Maturity or Age, Harvesting, Grading, Handling, Pre-freezing Treatment, Packaging, Freezing, Storage and Transport.

Investigations are being continued on the prefreezing treatment of cut fruits, including the inactivation of the oxidative enzymes by steam and sirup scalding, and by sulfur-dioxide penetration. The control of discoloration and ascorbic-acid loss in purees by deaeration, flash heating, and addition of antioxidants is being investigated. Basic techniques for the freezing preservation of several vegetables, some not commonly frozen, are being studied, for example tomatoes and soybeans. Critical investigations are under way on the factors influencing the quality of frozen peas. Studies are being made on emulsifying and foaming abilities and custard-baking quality as measures of frozen egg quality. The effect of several chemical and physical treatments on the quality of frozen egg yolk is being investigated.

RESEARCH PROJECT RRL-3-(3,5,7,8)-CP-6. - Investigations of the Microbiology and Histology of Apples, Fruits, Poultry Products and Vegetables Preserved by Freezing at Low Temperatures.

Investigations are under way on the value of propionate salts in suppressing growth of micro-organisms in fruits and vegetables intended for freezing. Specific findings prove that this compound is effective for the purpose indicated, and will retard spoilage of material unavoidably delayed, as during harvest gluts. A quick and easy test to gage the sanitary condition of broken-out eggs is being investigated. It depends on color changes in a solution of dye (resazurin) when a small sample of egg is added.

RESEARCH PROJECT RRL-3-(3,5,7,8)-CP-7. - Investigations of the Utilization of Apples, Fruits, Poultry Products and Vegetables Preserved by Freezing at Low Temperatures.

Considerable work has been done on the utilization of crushed and pureed fruit as sundae toppings, fruit-intrusion type of ice cream, jam manufacture, and as a flavor base for ice cream, sherbets, ices and beverages. These investigations are being continued. Two new products, utilizing frozen fruit juices and purees, have been developed and are being investigated further. These are Velva Fruit, a new frozen fruit dessert with a soft smooth texture, and a cold-processed



RESEARCH PROJECTS RRL-3-(3,5,7,8)-CP-7. (Continued)

gelled fruit product useful as a spread. Both of these products possess the natural flavor, color, and most of the nutritive value of the fresh fruit. Some preliminary studies have been made on a technique for the preparation of frozen vegetable purees by disintegration while in the frozen state.

Engineering and Development Division

RESEARCH PROJECT RRL-3-(3,5,6,7,8,9)-ED-6. - Engineering and Economic Investigations in the Industrial Processing of Western Agricultural Commodities for Food.

Dehydration and freezing as methods of preserving food commodities assigned to this Laboratory for investigation will be considered from two standpoints: industrial economics and engineering development. The supplies of commodities (fruits, vegetables, and poultry products) will be analyzed. Means will be sought to improve the operating effectiveness of steps in processing, and to improve equipment. Comprehensive engineering studies of vegetable dehydration have already been made; these will be continued and extended. Comparable investigations of industrial food freezing operations will be carried out.

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-ED-7. - Engineering and Economic Investigations in the Recovery of By-products from the Industrial Processing of Western Agricultural Commodities.

Laboratory processes for the recovery of by-products will be developed through a sequence of economic analysis, engineering investigation, and pilot-plant study. The objective is a satisfactory estimate of probable commercial utility. Processes for making a concentrated nutrient medium from waste asparagus butts and for growing yeast on fruit cannery waste have been investigated. Engineering studies of crucial process steps were made, pilot plants were set up and operated in cooperation with commercial firms, and suggestions for commercially practicable plants and processes are being drawn up for publication.

RESEARCH PROJECT RRL-3-(4)-ED-8. - Engineering and Economic Phases of the Development of High-Grade Alfalfa Feeds.

The economic opportunity for an expansion in industrial conversion of alfalfa to high-grade stock feeds will be investigated, and improvements in methods and equipment will be sought through engineering studies. Some investigations on the storage of alfalfa meal in inert gas in order the better to preserve its nutritive quality have already been made. A study of the equipment now being used for alfalfa dehydration, the cost of drying, and the potential field for expansion of alfalfa dehydration is now under way.



Physicochemical & Analytical Division

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9,10). --PA-4.- Macrochemical and Microchemical Analyses of the Agricultural Commodities and the Products Derived Therefrom.

The determination of the chemical constituents of agricultural materials and products and the development of new methods in order to expedite these determinations are essential in research and development work. Small quantities, 50 mg. or less, of soluble pectins or pectic acid in samples of fruits and vegetables have been measured; accurate and rapid measurements of moisture in starch, pectin, dehydrated eggs, vegetables, and related materials by the Karl Fischer reagent have been made; a rapid method for the determination of carotene in oil solutions without saponification has been developed in connection with work on extraction and stabilization of carotene from alfalfa.

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-PA-5. - X-ray, Microscopic, Crystallographic, Spectroscopic, Color, Electronic and Other Physical Methods of Testing and Analyzing Agricultural Materials and the Products Derived Therefrom.

A knowledge of the physical properties of agricultural materials and products is prerequisite to determinations of their industrial use and economic value. Surface area of dehydrated egg particles by the gas adsorption method has been measured; chemical microscopic studies of two germicides, lysozyme and citrinin, are under way; a spectroscopic method for the determination of trace amounts of lead in pectinous materials has been completed; and studies are in progress on the calcium content of vegetable juice residues and the copper in eggs. X-ray diffraction studies of egg albumin and pectin fibers are in progress. Color as a criterion of quality of fruit, vegetable and poultry products, and as a function of processing and storage, is under investigation.

RESEARCH PROJECT RRL-3-(3,4,5,6,7,8,9)-PA-6. - Colloidal, Chromatographic, Spectrophotometric, Fluorometric, Photochemical, Heat Transfer, Reaction Kinetics, and Other Physical Chemical Investigations Pertaining to the Agricultural Commodities and the Products Derived Therefrom.

Pilot-plant operations and commercial production of new or improved products are usually based on physical chemical data pertaining to the processes and products involved. The design of equipment and control of processing in the egg and vegetable dehydration industries are partly based on the moisture equilibrium data obtained as part of this project. Heat-transfer data on both dehydrated and fresh fruits and vegetables are being obtained for use in the dehydration and freezing of foods. Spectrophotometric and fluorometric methods are being used to study the causes of spoilage in egg powder, to establish an objective criterion of quality of dehydrated eggs, and to determine the carotene content of dehydrated vegetables.



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Protein Division

RESEARCH PROJECT RRL-3-(4,7,9)-P-3. - Chemical Composition, Reactions and Reactivity of Assigned Commodity Proteins.

Methods for the determination of cystine, cysteine and glutamic acid (both chemical and microbiological) have been developed and applied, along with established methods for other amino acids, in the examination of alfalfa, wheat, and poultry proteins. A method for the determination of the total acid and basic groups in proteins has been developed and applied. Progress has been made in our investigation of the mechanics of the protein-formaldehyde reaction and in the application of these and other fundamental data to the development of modified proteins for industrial application. The amine-sugar reaction, as related to the development of color, flavor, and odor in stored dried eggs has been investigated directly and by means of model systems, and application to changes in fruits and vegetables is anticipated.

RESEARCH PROJECT RRL-3-(4,7,9)-P-4. - Investigation of Structure and Structural and Physical Chemical Relationships in Alfalfa, Poultry, and Wheat Proteins.

Protein-detergent complexes have been prepared and their structure and composition studied by viscosity and electrophoretic measurements. Protein components of eggs have been similarly investigated. These and other protein derivatives and complexes will be investigated by ultracentrifugal measurements. Stress-strain and hysteresis curves for natural and synthetic protein fibers have been plotted from experimental data; the effect of treatment with urea (to weaken bonds between chains) and with cross-linking agents (to strengthen or establish bonds between chains) are being studied. Means for determining the effective chain lengths of linear polymers in fibers are under investigation.

RESEARCH PROJECT RRL-3-(4,7,9)-P-5. - Industrial Application and Utilization of Assigned Commodity Proteins.

Adhesives, fibers, films, paper coatings, plastics, and pharmaceutical preparations have been made from assigned commodity proteins and compared with established industrial products. Three of these (fibers, a modified phenolic plastic, and a gel-forming gluten derivative) have promising properties. As new protein derivatives with improved properties are developed, application in the proper fields will be made.



EASTERN REGIONAL RESEARCH LABORATORY

P. A. Wells, Director

Divisions and Division Leaders

Sym- bol	Division	Leader
A	Analytical and Physical Chemistry	M. J. Copley
B	Biochemical	J. J. Willaman
C	Carbohydrate	L. T. Smith
E	Chemical Engineering and Development	R. K. Eskew
H	Hides, Tanning Materials, and Leather	J. S. Rogers (actg.)
OF	Oil and Fat	W. C. Ault
P	Protein	R. W. Jackson

Commodities

- 4-3 Apples
- 4-4 Vegetables
- 4-5 Tobacco
- 4-6 Milk products
- 4-7 Animal fats and oils
- 4-8 White potatoes
- 4-9 Tanning materials, hides, skins, and leather

Research Projects

	Page
A-1 Analytical chemical investigations . . . . .	24
A-2 Physical chemical investigations . . . . .	24
A-3 Physical investigations . . . . .	25
A-4 Tobacco composition investigations . . . . .	26
B-1 Chemical research on apple utilization . . . . .	26
B-2 Chemical research on utilization of vegetable crops . . . . .	27
B-3 Chemical research on utilization of tobacco . . . . .	27
C-1 White potato starch and derived products . . . . .	28
C-2 Utilization of products derivable from lactose by fermentation and chemical degradation . . . . .	28
C-3 Modification and utilization of lactose . . . . .	29
E-1 Chemical engineering studies on the utilization of animal fats and oils, apples, milk products, tanning materials, hides, skins, and leather, tobacco, vegetable crops, and white potatoes . . . . .	29
E-2 Market surveys relative to and industrial development of processes arising from the utilization of animal fats and oils, apples, milk products, tanning materials, hides, skins, and leather, tobacco, vegetable crops, and white potatoes . . . . .	31
H-1 Hides and skins investigations . . . . .	32
H-2 Tanning materials and tanning processes investigations . . . . .	32
H-3 Leather investigations . . . . .	33
OF-1 Investigations on the chemical modification, excepting polymerization, of the unsaturated fat acids and their derivatives . . . . .	34



	Page
OF-2 Investigations on the utilization of animal fats and oils as raw materials for the production of surface active agents . . . . .	34
OF-3 Investigations on the fundamental chemistry of animal fats and oils and the technology of their production, processing, preservation, and use. . . . .	35
P-1 The conversion of milk proteins to commercially utilizable forms . . . . .	35
P-2 The production, purification, and characterization of the proteins of milk . . . . .	36
P-3 The chemical modification of milk proteins . . . . .	37
P-4 The composition and structure of pure proteins and derivatives. . . . .	37

Analytical and Physical Chemistry Division

RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-A-1. - Analytical Chemical Investigations.

Work on this project is conducted in a centralized Analytical Chemistry Section staffed with trained analysts and equipped with the most modern apparatus. The work is divided almost equally between research on analytical methods and the performance of standard analyses on materials used or produced by other units of the Laboratory. The analysis of the many new products constantly being produced has made it necessary to frequently devise new analytical procedures or modify existing methods. The variety of the constituents determined is illustrated by a partial list of new methods developed: a procedure for estimating hydroxyl groups in certain types of fatty materials; an assay method for rutin, a flavonol glucoside, in crude preparations; a new method for determining pectin in plants; several procedures for the analysis of plant extracts and latices for rubber; the estimation of  $\alpha$ -amino groups in insoluble proteins, and satisfactory procedure for determining salicylanilide (SHIRLAN) in leather mold-proofing solutions.

Several thousand analyses by standard procedures have been performed yearly on laboratory products. Many of these have required the use of specialized techniques and equipment. An increasing proportion of the total number of analyses are being performed by microchemical procedures.

RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-A-2. - Physical Chemical Investigations.

Work on this project is currently concentrated upon research on proteins, white potato starch, and apple pectin.

Emphasis in the protein work has been placed on development of methods for improving the quality of fiber made from globular proteins by changing the shape of the protein molecules and the manner in which they are associated in the fiber. Through appropriate chemical and mechanical



RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-A-2. (Continued)

treatment it has been possible to give the globular molecules an extended linear form, and to pack these in a regular spatial array. As revealed by X-ray diffraction the orderliness of arrangement of the modified molecules is high, approaching that in the natural fibers, silk and flax. By these treatments the tensile strength and the dimensional stability are pronouncedly increased. Means are being sought for enhancing the flexibility and resiliency of the fiber, for in these respects it is still deficient.

Investigation of white potato starch was recently begun with a study of the viscosity stability of pastes made from white potato starch modified by heat and moisture. Work to be undertaken includes correlation of properties of starch solutions, gels, pastes and films with the properties of starch granules, molecules and molecular aggregates which enter into their composition.

In the pectin research, a study is being made of the important factors that determine the jellying and thickening power of natural apple pectin and especially the "low ester" pectins produced from natural pectin by enzyme- and acid-catalyzed demethylation. Among the factors are molecular weight, degree of demethylation, solubility, pH and concentration of metal ions. Studies of the rate and mechanism of the demethylation process and the mechanical behavior of pectin gels are being carried on also.

RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-A-3.- Physical Investigations.

The work under this project is conducted in a centralized Physics Section prepared to perform highly specialized research, analysis, and testing involving the application of physics to the chemical and technological program of the Laboratory. Specialized equipment includes a Beckman ultraviolet spectrophotometer, a General Electric automatic recording spectrophotometer for the visible spectrum, X-ray diffraction apparatus, electron microscope, microscopic equipment, and various physical testing instruments for determining the mechanical, electrical, and optical properties of materials.

The important investigations under way at present are:

- (1) Development of improved and extended ultraviolet spectrophotometric methods for the analysis of fats and soaps for small proportions of polyunsaturated constituents, in cooperation with the Rubber Reserve Company.

- (2) Research on the mechanism of oxidation and development of rancidity in animal fats. Ultraviolet and infrared absorption methods are being applied.

- (3) The development of improved casein fibers, by conversion of the protein molecules to a linear oriented configuration to improve their wet and dry tenacities. X-ray diffraction, birefringence studies, and electron microscopy are applied to this investigation.



RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-A-3.(Continued)

(4) The evaluation of allyl starch as a material for use in lacquers, adhesives, and laminated plastics.

(5) The investigation of protoplasts from vegetable waste materials such as broccoli leaves as a plastic molding material.

(6) Color studies for purposes of quality evaluation, on leather and tanning materials, tobacco, potato starch, and lard.

(7) Physical testing and evaluation of casein plastics, elastomers of methyl acrylate, and rubber extracted from *Cryptostegia*.

(8) Miscellaneous minor investigations, such as characterization of purified compounds by X-ray diffraction and spectrophotometry, investigation of special colorimetric chemical analyses, particle size determinations with the electron microscope.

RESEARCH PROJECT RRL-4-(5)-A-4. - Tobacco Composition Investigations.

During the past year, attention has been concentrated on the chemistry and clinical applications of rutin, the flavonol glucoside isolated from flue-cured tobacco. The medical reports continue to be favorable. As a result of published reports widespread interest in the medical applications of rutin has been aroused. It has been furnished to a large number of physicians for treatment of conditions associated with capillary fragility. These include its use by ophthalmologists for certain eye conditions and its application in obstetrics for treating patients having complications arising from the Rh factor.

The chemical studies on rutin have revealed the fact that the buckwheat plant is a much better source of rutin than is cured tobacco. Studies were made on the optimum stage of growth for maximum rutin yield, methods for handling the green plant to minimize losses of rutin, and processes for extracting and purifying the glucoside from buckwheat. Seven tons of green buckwheat were processed during the summer of 1944 and approximately twenty pounds of pure rutin were prepared. Part of this supply was manufactured into 250,000 tablets, of 20 mg. rutin each, for purposes of administration to patients with increased capillary fragility.

Biochemical Division

RESEARCH PROJECT RRL-4-(3)-B-1.- Chemical Research on Apple Utilization.

Since low-methyl pectins offer the possibility of new types of fruit and vegetable products, such as jellies of low sugar content and fruit and vegetable aspics and salads, the preparation of these special pectins, called pectinates, has been studied in detail. Of the two methods used here, acid hydrolysis gives a product of better gelling properties, but enzyme hydrolysis would be much better from the manufacturing viewpoint, and for some purposes either product would be suitable. Details of each



RESEARCH PROJECT RRL-4(3)-B-1. (Continued)

method have been worked out, including the use of polyphosphates for extracting pectin; the properties and sources of enzyme; the addition of materials for facilitating drying of the final product.

A simple process has been developed for producing apple sirup, mainly for industrial purposes. This has been in commercial use for three seasons. Somewhat different processes have now been developed for producing table or food sirups. Malic acid may be a by-product from one of these.

Both good and poor lots of dried apple pomace have appeared on the market for use in pectin manufacture. An attempt is being made to discover the factors involved in pomace quality and to bring them under control. If this succeeds, apple pomace can better meet the competition of citrus pulp as a source of pectin.

RESEARCH PROJECT RRL-4-(4)-B-2. - Chemical Research on Utilization of Vegetable Crops.

Attention has been given almost exclusively to the leafy wastes of the vegetable industry, partly because they have so little economic value now, partly because we have found them to be surprisingly rich sources of protein, carotene, and riboflavin. Both experimental and practical feeding trials show that at least six of them are superior to the average present day alfalfa leaf meal in poultry rations. These are beet, spinach, carrot, turnip, lima, and broccoli leaf meals.

Some of these leaf meals, notably broccoli, could be a source of commercial carotene, since many samples of it have the highest carotene content of any crop examined. Methods have been developed for the extraction and purification of this carotene, leading either to the crystalline product or to a concentrate suitable for feed or pharmaceutical use.

RESEARCH PROJECT RRL-4-(5)-B-3. - Chemical Research on Utilization of Tobacco.

From the beginning, we have concentrated on nicotine, with the objectives of (1) improving its recovery from tobacco, (2) studying its conversion to nicotinic acid, (3) furthering its use as an insecticide.

Not much has been accomplished on nicotine recovery because of the lack of personnel. Processes have been developed and published for both liquid phase and vapor oxidation of nicotine to nicotinic acid, and for the recovery of the latter from its solutions. Nicotinic acid esters and amides of high molecular weight, and hence of low solubility in water, have been developed and are being tested for the fortification of cereal foods which are rinsed prior to cooking.



RESEARCH PROJECT RRL-4-(5)-B-3. (Continued)

Several series of nicotine derivatives have been prepared which are new to science. One of these series is showing promise in various insecticide combinations, another has high fungicidal activity, and a third is being tested pharmacologically.

Carbohydrate Division

RESEARCH PROJECT RRL-4-(8)-C-1. - White Potato Starch and Derived Products.

Investigations on starch derivatives have resulted in the development of a method for the preparation of a soluble allyl ether of starch. The gummy allyl starch prepared by this method is soluble in a number of organic solvents and can be applied as a coating on wood, paper, textiles, metal and glass. It produces a smooth glossy film which on heating or in the presence of catalysts becomes resistant to the action of organic solvents, oils, acids and alkalis, and to heat up to 200° C. The commercial possibilities of allyl starch as protective and decorative coatings and as adhesives and plastics are being investigated. Dextrinization of potato starch has been studied and methods of preparation of starch acetates have been improved.

Studies are in progress on the physical and chemical properties of potato starch looking to the establishment of a rational basis for new and expanded industrial uses of this product. Changes in the properties of potato starch during storage as well as possible improvements in manufacturing methods will be investigated.

RESEARCH PROJECT RRL-4-(6)-C-2. - Utilization of Products Derivable from Lactose by Fermentation and Chemical Degradation.

The work thus far has been centered upon lactic acid, which is manufactured from lactose in whey in high yield by fermentation.

The increased utilization of lactic acid and its derived products was seriously handicapped by the lack of a satisfactory method of isolating pure lactic acid from the fermentation liquor. This handicap was successfully overcome by the development of a convenient and efficient method of preparing either pure lactic acid or lactic esters directly from the fermentation liquor.

Lactic acid has been converted in high yields into plasticizers and polymerizable allyl derivatives. The plasticizers have potential use in cellulose and vinyl plastics and in synthetic rubbers; when polymerized the allyl derivatives of lactic acid yield colorless, transparent, glass-like resins.

Efficient and continuous methods for transforming lactic acid into acrylic esters were developed. Polymerization of the acrylic esters yields resins having a wide range of properties. Some of the resins are hard,



## RESEARCH PROJECT RRL-4-(6)-C-2. (Continued)

colorless, and transparent, whereas others are soft and rubbery. By compounding and vulcanizing the soft acrylic resins, a synthetic elastomer that is superior to natural rubber in some respects is obtained. This new elastomer is called Lactoprene to indicate derivation from lactic acid.

## RESEARCH PROJECT RRL-4-(6)-C-3. - Modification and Utilization of Lactose.

Due to initial limitations of staff and subsequently to the urgency of other phases of the research program, no work has been conducted on this project. It is planned to initiate investigations soon on the development of improved methods for the production of lactose. Attempts will be made to adapt modern methods of cane sugar manufacture to reduce the cost of making lactose. Derivatives of lactose will be synthesized and evaluated for pharmaceutical and other industrial applications.

Chemical Engineering and Development Division

RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-E-1. - Chemical Engineering Studies on the Utilization of Animal Fats and Oils, Apples, Milk Products, Tanning Materials, Hides, Skins and Leather, Tobacco, Vegetable Crops, and White Potatoes.

Animal Fats and Oils

Modifications have been made in the design of a pilot plant high-vacuum, high-temperature distillation unit to permit chemical engineering investigations on the commercial feasibility of preparing a highly purified oleic acid from animal fats.

Apples

A pilot plant unit with an evaporator of 100-gallon capacity was designed and has been operated for three years in the experimental production of sirups and concentrates from apple juice.

A continuous process for the recovery and concentration to an essence of the volatile flavoring constituents of apple and other fruit juices has been developed and operated on a pilot plant scale. Apple essence when returned to the concentrated juice results in a full-flavor concentrated product from which a juice can be reconstituted by the addition of water to form a beverage indistinguishable in taste and aroma from fresh apple juice. Full-flavor concentrate can also be used in ices, bakery products and in compounding table sirups.

Pilot plant scale studies on the drying of apple pomace are being carried out to determine the conditions of drying which result in the highest grade of pectin recoverable from the pomace.



RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-E-1. (Continued)

Milk Products

A flexible pilot plant experimental esterification and distillation unit has been designed and ordered for the preparation of acrylic esters from crude lactic acid--derivable from milk by fermentation of the lactose.

A glass-lined reaction kettle, reflux condenser and accessories have been assembled for the pilot plant production of acrylic ester polymers and copolymers. Such products are used for coating textiles, cloth and other materials and when copolymerized with unsaturated compounds can be vulcanized to form products with valuable rubber-like properties.

Tanning Materials, Hides, Skins and Leather

Chemical engineering studies were made to determine the most economical commercial means for drying canaigre roots to produce a good grade of tannin. Approximately 2700 pounds of sliced roots were dried and ground for commercial tanning tests.

Pilot plant studies were made on the separation of stems and leaves on several varieties of sumac. Approximately 4,000 pounds of a leaf product were separated and ground for industrial evaluation as a source of tannin.

Tobacco

Laboratory studies showed that certain types of tobacco contained the drug rutin, of great value in reducing tendency towards cerebral hemorrhage. Rutin was found to occur in much larger amounts in buckwheat. Chemical engineering studies were carried out on a pilot plant scale to determine commercial means for the production of rutin from buckwheat and 20 pounds (enough for about 400,000 doses) of highly purified rutin were produced.

Vegetable Crops

Chemical engineering studies and pilot plant operations have been carried out on the preparation of high-vitamin, high-protein leaf meals from broccoli waste, pea vines, lima bean vines and the tops of beets, carrots and turnips. Large quantities of meals from several of these wastes have been prepared for feeding tests and market evaluation.

Potatoes

A monel reaction kettle with accessories has been designed and ordered for the pilot plant experimental production of allyl starch. Allyl starch can be used for chemical- and wear-resistant coatings.



RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-E-2. - Market Surveys Relative to and Industrial Development of Processes Arising from the Utilization of Animal Fats and Oils, Apples, Milk Products, Tanning Materials, Hides, Skins and Leather, Tobacco, Vegetable Crops, and White Potatoes.

#### Animal Fats and Oils

A preliminary market survey has been made to determine the potential market and price of a highly purified oleic acid developed on a laboratory scale from animal fats. This showed that there was a potential market for a higher grade of oleic acid and that a product of the quality produced in the laboratory might be expected to command a price from one to two cents above that of the present best grade of refined red oil.

#### Apples

Industrial development on apple juice products resulted in the use by the tobacco industry of several million pounds annually of bland apple sirup as a conditioner.

Samples of apple juice products have been widely distributed to the tobacco, confectionery, bakery, cosmetic, pharmaceutical, cork and other industries and arrangements have been made for industrial evaluation of these products. The use of apple juice products has been furthered through contact with associations of apple producers and sirup manufacturers as well as through industries where the sugar content or plasticizing effect of apple sirup were applicable.

#### Tobacco

Because of the decreased supplies of rotenone, arrangements were made with federal and state agencies for the field evaluation of insecticide dusts, employing nicotine alkaloid and sulfur in part substitution for rotenone. These tests showed that the required amount of rotenone was reduced by one-half or more while the efficiency of control was maintained or increased. This development has pointed the way for a new series of studies on synergism where nicotine is used with DDT.

Cooperative orchard tests were made with federal, state and industrial agencies for the evaluation of a series of double salts of nicotine prepared experimentally at the laboratory. Several of the compounds show promise. Arrangements have also been made to evaluate some of these compounds as fungicides.

It was shown that the use of a mixture of nicotine bentonite with other materials required for insecticide purposes was effective in repelling rabbits.



RESEARCH PROJECT RRL-4-(3,4,5,6,7,8,9)-E-2. (Continued)

Vegetable Crops

A comprehensive survey was made on the availability and seasonal distribution of vegetable wastes suitable for the production of high-protein, high-vitamin leaf meals. A survey was also made in a section of Florida in which there is available a large concentration of vegetable wastes as well as a need for cattle and poultry feed.

Hides, Tanning Materials, and Leather Division

RESEARCH PROJECT RRL-4-(9)-H-1. - Hides and Skins Investigations.

Hides and skins are essential for making leather, which is now rated as being the seventh most important critical material. They can be conserved by careful handling and effective curing. Results of investigations looking toward their preservation and improvement have been recorded in 46 publications issued between 1919 and 1943. They deal with: improved methods for skinning, curing and handling; damages resulting from follicular mange, ringworm, non-parasitic eczema and nematode; salt tolerant bacteria; reddening of hides; and inefficient curing resulting from use of old, bloody salt. They show: that concentrations of salt in hides above three molar stimulate growth of halophilic bacteria; that moisture contents between 49 and 55 percent are critical as regards spoilage of salted calfskins; and that full feeding and underfeeding influence the character of lambskins and the quality of the leather they produce. The addition of various chemicals, such as para nitrophenol and sodium silicofluoride, to curing salt has resulted in increased preservative action. Investigations now in progress include: studies of effectiveness of various curing procedures; determination of the effect of chemicals added to curing salt upon the tensile strength of the leathers produced from the specially cured hides; development of a chemical method for determining degree of spoilage in hides; and studies of the effect of various animal life factors, such as breed, feed and vitamin deficiency upon hides and skins and the leather produced from them.

RESEARCH PROJECT RRL-4-(9)-H-2. - Tanning Materials and Tanning Processes Investigations.

This country now produces less than one-third of the tannins that it uses for making vegetable-tanned leathers, and because its most important domestic source, the chestnut tree, has been killed by blight, it will become increasingly dependent upon imported tannins unless additional domestic supplies are developed. Investigations conducted include: studies of woods-peeled western hemlock bark, making experimental tanning extract and its successful use in semicommercial heavy leather tanning tests; determination of tannin content of domestic sumacs of eastern United States; experimental cultivation, curing and handling of sumac in cooperation with Bureau of Plant Industry, Soils and Agricultural Engineering, and Soil Conservation Service; determination by actual tanning tests that properly handled domestic leaf compares quite



RESEARCH PROJECT RRL-4-(9)-H-2. (Continued)

favorably with imported sumac; cooperative field tests with Bureau of Plant Industry, Soils and Agricultural Engineering on growing canaigre in Texas, New Mexico and Arizona that show under favorable conditions calculated root yields up to 10 tons per acre; laboratory studies showing average tanning contents of dry canaigre roots to be about 20-25 percent, that excess sugars can be removed by cold water and tannin subsequently by warm water, but that at high temperatures starch interferes; and laboratory preparation of canaigre extract and its successful use alone and blended with other tannins in heavy leather tanning tests. Work now under way includes: continued cooperative studies on canaigre and sumac; selection of high tannin strains for cultivation; increased field production of canaigre; preparation of bulletin on domestic sumacs; further studies on curing, effects of heating and drying on quality, and comparison of tanning properties of different domestic sumacs; continued studies for improvement of methods for leaching canaigre; preparation of tanning extracts and economical utilization of canaigre starch and sugar; and a project for the development of Florida scrub oak bark conducted in cooperation with the Engineering and Industrial Experiment Station of the University of Florida.

RESEARCH PROJECT RRL-4-(9)-H-3. - Leather Investigations.

Leather, now a critical war material, is a national necessity at all times. Investigations, having as their objectives the evaluation of quality and serviceability of leather and the development of better leathers for special purposes, have been in progress for many years. Some of the studies conducted which are of particular interest include: development of methods for analysis and physical testing; first extensive organized wearing tests conducted with soldiers and civilians to determine relative wear resistance of different kinds of sole and upper leathers and of leathers from different parts of the hide; acquiring comprehensive data on composition of American sole leathers; development of preservative and waterproofing treatments for leather; demonstration that deterioration of bookbinding leathers is primarily due to acid pickup from polluted atmospheres; development of standard gas chamber for accelerated aging tests on leather and as an aid in evolving leathers resistant to decay; demonstration that chrome, chrome retanned vegetable, and alum retanned vegetable leathers are more resistant to acid rot than vegetable tanned leathers; determination of physical properties of alum retanned vegetable leathers, obtaining results showing that these leathers compare with chrome leathers in their resistance to moisture at elevated temperature, and suggesting that they might be used to replace chrome leathers in event of a chrome shortage; development in cooperation with the Tan-ners' Research Laboratory of a tentative method for evaluating effectiveness of fungicidal treatments for leather; and development, at the request of the Ordnance Department, of methods and a treating compound for rendering leather carrying cases moisture and mold resistant, and for rendering leather knife handles resistant to staining, molds, and embrittlement. Work now in progress includes: continuation of studies on alum retanned vegetable leathers, particularly as related to shoe upper and insole leathers; studies requested by Ordnance Department



RESEARCH PROJECT RRL-4-(9)-H-3. (Continued)

of various volatile fungicides with the object of selecting an effective fungicide and developing a method of application for the successful protection of optical lenses against fungal growth; and development of equipment suitable for measuring the resistance of upper leather to scuffing, and of sole leather to abrasion.

Oil and Fat Division

RESEARCH PROJECT RRL-4-(7)-OF-1. - Investigations on the Chemical Modification, excepting Polymerization, of the Unsaturated Fat Acids and their Derivatives.

Methods have been developed in the laboratory which offer considerable promise of being useful in large scale commercial production of oleic acid of relatively high purity from animal fats and oils. The process consists essentially of selective hydrogenation of the fats to produce the linoleic acid content below 1%. This is then followed by conventional hydrolysis and pressing or preferably by hydrolysis and crystallization from a solvent such as acetone. Removal of the linoleic acid eliminates the necessity for actual crystallization of the oleic acid.

Work on the preparation of chemical derivatives of oleic acid, particularly those obtained by oxidative reactions, has been intensified. Considerable advances have been made in the preparation of epoxystearic acid. Moreover it has been discovered that when 30% hydrogen peroxide is added to a mixture of formic acid and oleic acid, very little available oxygen is lost. Since this makes possible the oxidation of oleic acid with an equimolecular quantity of hydrogen peroxide to give 9,10-dihydroxystearic acid practically quantitatively, it is hoped that this discovery will furnish the basis for an industrial process for the production of this latter compound.

RESEARCH PROJECT RRL-4-(7)-OF-2. - Investigations on the Utilization of Animal Fats and Oils as Raw Materials for the Production of Surface Active Agents.

An investigation of tallow soaps for use as emulsifiers in the production of GR-S synthetic rubber carried out in cooperation with Rubber Reserve Company has been essentially completed. It seems probable that the presence of, and variations in, polyunsaturated constituents in the emulsifiers are to a high degree responsible for retarding, and variations in retarding, the polymerization of GR-S synthetic rubber. Mild selective hydrogenation of the tallows used in the preparation of the soaps is indicated as the best remedy for the present difficulties in the process.

We are at present engaged in the large scale laboratory preparation of certain fatty acid derivatives which are of considerable interest to the Naval Research Laboratory for certain uses which are confidential at present.



RESEARCH PROJECT RRL-4-(7)-OF-3. - Investigations on the Fundamental Chemistry of Animal Fats and Oils and the Technology of their Production, Processing, Preservation and Use.

Considerable work is being done on the comparison of different rapid methods for determining the stability of fats both in the presence and absence of a large number of added antioxidants of several different chemical types. Moreover, the results obtained by the rapid methods are being compared with stability as measured by actual storage. When more data become available it may be possible in most instances to predict more accurately the storage life of shortening from the stability as determined by a rapid method.

Results obtained in the investigation of oxygen absorption of pure fatty components of lard have shown that the ease of oxidation of a fat in the absence of antioxidants is dependent upon the presence and percentage of polyunsaturated fatty constituents. The results explain why selective hydrogenation of fats increases resistance to oxidative rancidity to a greater extent than non-selective hydrogenation.

Work on synthesis and evaluation of antioxidants suitable for use in animal fats is being continued. A cooperative research program of considerable magnitude with the Subsistence Research & Development Laboratory of the U. S. Army Quartermaster Corps is being prosecuted.

Protein Division

RESEARCH PROJECT RRL-4-(6-P-1). - The Conversion of Milk Proteins to Commercially Utilizable Forms.

Casein Bristle Fiber

A bristle fiber has been developed from casein which is suitable for paint brushes and other types of brushes. The fiber is made by extruding casein containing from 30 to 40 percent water through an appropriate die heated to 200°-205° F. The fiber is made black, more durable and stronger by stretching in an aqueous quinone solution. Paint brushes made from casein bristles are satisfactory for use with oil paints, being resistant to organic solvents. However, they soften when soaked with water and are not suitable for use with water paints. Casein bristle brushes pick up and carry paint well, are resistant to abrasion and make a smooth film. The casein bristle fiber is approximately two-thirds as strong as natural bristle and is less tough. Further experimentation is directed toward the improvement of the casein fiber and its commercial development. Two companies have obtained permission for making casein bristles by our method.

Casein Textile Fiber

A very large potential use for proteins lies in the development of a more satisfactory synthetic textile fiber, a "protein rayon". Such work is being intensively pursued in laboratories throughout the world, and results to date indicate that an industry of large magnitude



RESEARCH PROJECT RRL-4-(6)-P-1. (Continued)

will develop. In this laboratory we have produced a casein fiber which is stronger than similar fiber now on the market. Supplementary studies of dyeing, acetylation and other finishing treatments, physical properties and test methods, and internal structure as shown by X-ray have been carried out which have given us chemical and physical knowledge of the fiber. This knowledge enables us both to anticipate its performance under a wide variety of conditions and to direct our work, especially toward further enhancement of the wet strength.

Plastics

Improvements in the molding characteristics and dimensional stability of plastics made from formaldehyde-treated casein and casein otherwise modified (see project above) have been obtained. For example, a molding powder formulated with a suitable type of organic plasticizer has given satisfactory moldings which are dimensionally stable to ordinary atmospheric conditions. The tensile strength is not decreased as when additional water is used, and the water absorption is curtailed.

RESEARCH PROJECT RRL-4-(6)-P-2. - The Production, Purification, and Characterization of the Proteins of Milk.

Composition and Fractionation of Casein

A knowledge of the composition of proteins is necessary for a systematic investigation of the various aspects of their utilization. Studies were accordingly undertaken on the protein components of which casein is composed and their separation. It was found possible to separate casein by a chemical procedure into two fractions which correspond to the two components indicated by electrophoretic analysis. These two fractions,  $\alpha$ - and B-casein, are not homogeneous proteins, but each consists of more than one component. Neither of these fractions offers sufficient advantages over the unfractionated casein to justify using the complex fractionation procedure on a large scale basis.

An important result of this investigation was the detection of a proteolytic enzyme in casein. This enzyme is present in all preparations of commercial and highly purified casein and causes its slow hydrolysis in solution. The information obtained on the behavior and properties of the enzyme are essential in defining the optimum conditions in any industrial process employing casein.

A simple and economical method has been devised for preparing casein on a small scale for commercial purposes.

Whey Protein

The use of precipitating agents in the recovery of proteins from cheese whey has been studied. It was found that organic sulfates and sulfonates are excellent precipitating agents for whey proteins. A simple method was devised for preparing cellulose sulfate. Whey protein recovered by



RESEARCH PROJECT RRL-4-(6)-P-2. (Continued)

means of cellulose sulfate is water soluble and heat coagulable. It was found that glues and fibers prepared from whey proteins were inferior to corresponding products prepared from casein.

RESEARCH PROJECT RRL-4-(6)-P-3. - The Chemical Modification of Milk Proteins.

Casein has been modified chemically by a variety of reactions to decrease its affinity for water and thus to enhance its usefulness for making plastics and fiber. Extensive investigations of the acylation of casein and of the modification of the protein by cyanate treatment have yielded improved casein molding powders. Thus, strong plastic articles with materially increased resistance to water and concomitant dimensional stability have been molded directly from casein modified by the introduction of higher fatty acid residues. Unusually strong plastic objects with relatively low water absorptions have been made by direct molding of cyanated casein. In contrast to such molding of the finished article, the commercial process in use today for manufacture of protein plastics is more complicated, involving hardening of the shaped article in a formaldehyde bath.

A quantitative investigation of the benefits to be derived from the acetylation of spun casein fiber has established conditions whereby increased resistance to boiling solutions as well as reduced affinity for dyes may be attained.

RESEARCH PROJECT RRL-4-(6)-P-4. - The Composition and Structure of Pure Proteins and Derivatives.

A principal portion of this program, an exact study of the amino acid composition of pure proteins, has been held in abeyance during the war. One phase which has been investigated is the effect of protein structure and derivatives upon water absorption. Thus, casein as well as other proteins, chemically modified proteins, protein fibers, protein plastics and amino acids (protein units) have been subjected to extensive tests. An important study of the hydration of B-lactoglobulin crystals was completed.







NORTHERN REGIONAL RESEARCH LABORATORY

H. T. Herrick, Director

Divisions and Division Leaders

Sym- bol	Division	Leader
A	Analytical and Physical Chemical	R. T. Milner
CD	Commodity Development	J. H. Shollenberger
E	Engineering and Development	C. T. Langford
F	Fermentation	R. D. Coghill
MF	Agricultural Motor Fuels	C. T. Langford (actg.)
OP	Oil and Protein	J. C. Cowan
R	Agricultural Residues	E. C. Lathrop
SD	Starch and Dextrose	G. E. Hilbert

Commodities

- 5-3 Agricultural residues
- 5-7 Corn, wheat, and other cereal crops
- 5-8 Soybeans and other oilseed crops

Research projects

	Page
A-1 Analytical chemical investigations . . . . .	39
A-2 Physicochemical and physical investigations . . . . .	39
CD-1 Collection, preparation, and storage of materials required for the research work of the Laboratory and the integra- tion of the developmental work of the Laboratory with the breeding, selection, and production work of Federal, State, and private agencies. . . . .	40
CD-2 Agricultural, industrial, and market surveys for commo- dities, products, and processes. . . . .	40
E-1 Engineering and pilot plant development. . . . .	41
E-2 Processing of cereal grains on a pilot plant scale . . . .	41
F-1 The maintenance of a collection of industrially important yeasts, molds, and bacteria. . . . .	41
F-2 A study of the natural history and variability of indus- trially important micro-organisms. . . . .	42
F-3 A survey of the fermentative activities of individual or- ganisms contained in the culture collection of indus- trially important yeasts, molds, and bacteria. . . . .	42
F-4 The development of industrial fermentations using corn, wheat, and other cereal crops or products derived from them as substrates . . . . .	42
F-5 The development of industrial fermentation processes using agricultural residues or products derived from them as substrates . . . . .	43
F-6 The isolation, identification, and utilization of chemi- cals produced by fermentation. . . . .	43
MF-1 Pilot scale technological investigations of the production of agricultural motor fuels and byproducts by fermenta- tion . . . . .	43



	Page
MF-2 Formulation, development, investigation, and testing of gaseous, liquid, and solid motor fuels from agricultural materials. . . . .	44
MF-3 Laboratory-scale technological investigations of the production of motor fuels and byproducts from agricultural materials. . . . .	44
OP-1 Composition and processing of soybeans and other oilseeds, and of corn and other cereal grains for oils and proteins. . . . .	44
OP-2 Modification of linseed, soybean, and other vegetable oils of the Northern region and the components thereof. . . . .	45
OP-3 Polymerization reactions of soybean and linseed oils, their fat acid components and derivatives. . . . .	45
OP-4 Soybean, linseed, corn, and other protein investigations .	45
R-1 Development of papers and pulps from agricultural residues	46
R-2 Development of building materials and miscellaneous products from agricultural residues. . . . .	46
R-3 Development of plastics and plastic-like materials from agricultural residues. . . . .	46
R-4 Investigation of chemical constitution, reactions, and physical properties of lignin obtained from agricultural residues . . . . .	47
R-5 Development of industrial uses for hemicelluloses and derivatives from agricultural residues . . . . .	47
SD-1 Starch granule formation, properties, and composition. . .	48
SD-2 Starch structure, properties, and hydrolysis . . . . .	48
SD-3 Starch: chemical conversion . . . . .	48
SD-4 Dextrose: modification, condensation, and polymerization.	49
SD-5 Dextrose: rearrangement and degradation . . . . .	49

### Analytical and Physical Chemistry Division

#### RESEARCH PROJECT RRL-5-(3,7,8)-A-1. - Analytical Chemical Investigations.

The chemical composition of representative samples of corn, wheat, sorghum, sunflower, and safflower have been determined to furnish information for the research of the Laboratory, and to evaluate the influence of climate and variety on composition and hence on ultimate use of the crops. Improved methods for determining starch in cereals have been studied and adopted. Improvements in the analysis of soybeans for oil, in the determination of moisture in cereals and oilseeds, and in the analysis of oils are being studied. A large number of research analyses are carried out for the Laboratory as a whole.

#### RESEARCH PROJECT RRL-5-(3,7,8)-A-2. - Physicochemical and Physical Investigations.

Physical methods of analysis have been used on penicillin and its derivatives, starch, oils, and many other materials of importance in the research program of the Laboratory. Absorption spectrophotometry in the ultraviolet and X-ray diffraction pictures have been of service,



RESEARCH PROJECT RRL-5-(3,7,8)-A-2. (Continued)

particularly in identifying penicillins, starches, and oils. Equipment for absorption spectrophotometry in the infrared region has been ordered and will be used in studying oils and identifying products from the saccharification of agricultural residues.

Commodity Development Division

RESEARCH PROJECT RRL-5-(3,7,8)-CD-1. - Collection, Preparation, and Storage of Materials Required for the Research Work of the Laboratory and the Integration of the Developmental Work of the Laboratory with the Breeding, Selection, and Production Work of Federal, State, and Private Agencies.

Records are maintained and monthly reports are made on the current production, supply, and utilization situation of wheat, corn, and other cereal grains, and of soybeans and other oilseeds. New industrial developments on the utilization of these commodities and related commodities are reported, special emphasis being directed toward the productive capacity and the source materials for the alcohol industry as a whole. The economics of the production and utilization of starch and gluten from wheat and wheat flour are being studied.

Statistical investigations are being made of data resulting from organoleptic testing of oils, and of data resulting from penicillin assays.

RESEARCH PROJECT RRL-5-(3,7,8)-CD-2. - Agricultural, Industrial, and Market Surveys for Commodities, Products, and Processes.

Agricultural materials used for special experiments are obtained as needed. Besides these, series of samples are obtained for general physical and chemical analyses. The data thus obtained are compiled, studied, and made available for formulation of plant breeding aims and their integration with quality requirements for industrial utilization purposes. These series include:

Wheat and corn samples representative of commercial grades;

Waxy varieties of all the different cereals;

Varietal and environmental samples of corn, wheat, sunflower seed, and sorghum grain;

Samples of three series of single cross corn hybrids grown at state experiment stations; and

Samples of various kinds and varieties of agricultural residues.



### Engineering Division

#### RESEARCH PROJECT RRL-5-(3,7,8)-E-1. - Engineering and Pilot Plant Development.

Research conducted under this project is entirely in the field of chemical engineering and is concerned with the development of processes originating in the research divisions of the Laboratory. It is necessarily a comprehensive project, including industrial analysis, pilot plant testing, and engineering design work on nearly every type of problem studied in the entire Laboratory. A recent accomplishment which typifies the scope of the project was the pilot plant development of a process for recovering butylene glycol from fermentation liquors. At present, the problems under investigation include the fractionation of vegetable oils and derived products by the use of liquid-liquid extraction and the production of saccharic acid from dextrose.

#### RESEARCH PROJECT RRL-5-(7)-E-2. - Processing of Cereal Grains on a Pilot Plant Scale.

Starch and accompanying byproducts of known history are prepared from cereal grains on a pilot plant scale as required for research purposes in other divisions of the Laboratory. Pilot plant experiments on the processing of cereal grains for the purpose of developing new and improved methods of operation are also carried out. Recently a new process has been developed for the production of wheat starch from whole wheat. Problems under investigation at the present time include the fractionation of starch into amylose and amylopectin, the production of undevitalized wheat gluten, and a determination of the optimum conditions for producing steepwater to be used in the manufacture of penicillin.

### Fermentation Division

#### RESEARCH PROJECT RRL-5-(3,7)-F-1. - The Maintenance of a Collection of Industrially Important Yeasts, Molds, and Bacteria.

For extensive work in the general field of fermentation, a good collection of micro-organisms having industrial significance is essential. Such a collection was established early in the history of this Laboratory, and at present is composed of approximately 2,000 strains of molds, 1,000 of yeasts, and 600 of bacteria. This collection gradually increases in size, but no attempt is being made to add to it extensively, except in the cases of specific organisms such as Penicillium notatum and Aspergillus terreus, which are of immediate concern to us. The collection is carried on conventional agar slants, but in addition is completely lyophilized in triplicate. This collection contains practically no pathogenic organisms.

Cultures are sent on request (at no charge) to other Government agencies, State Experiment Stations, and university and industrial research workers to whom we are under obligations or who are working with processes developed at this Laboratory. In general we prefer, whenever



RESEARCH PROJECT RRL-5-(3,7)-F-1. (Continued)

possible, to deposit cultures which are in demand with the American Type Culture Collection and refer requests to them. There is a free interchange of cultures between these two collections.

RESEARCH PROJECT RRL-5-(3,7)-F-2. - A Study of the Natural History and Variability of Industrially Important Micro-organisms.

A successful industrial fermentation is dependent primarily on the quality of the microorganism which brings it about. Wild strains of a species, isolated at random from natural sources, vary enormously in the efficiency with which they carry out a given reaction. In addition to conducting extensive surveys for the purpose of finding the best organism available for a given purpose, it has been found advantageous to take the best wild strains available and improve them by processes of selection or by deliberately bringing about variation by means of such devices as ultra-violet irradiation, X-ray treatment, or hybridization (when possible).

Procedures of this nature have been used successfully by the Fermentation Division in the development of improved strains of Aspergillus terreus for the itaconic acid fermentation, and they are proving to be of great value in the development of improved strains of Penicillium notatum and Penicillium chrysogenum for use in the production of penicillin. Variations of these methods will continue to be used as new fermentation processes are studied.

RESEARCH PROJECT RRL-5-(3,7)-F-3. - A Survey of the Fermentative Activities of Individual Organisms Contained in the Culture Collection of Industrially Important Yeasts, Molds, and Bacteria.

The Culture Collection of the Fermentation Division contains hundreds of microorganisms whose potentialities in the field of fermentation are completely unknown. There is no question but that a thorough survey will uncover new metabolic products which will find uses in the chemical industry and can be commercially exploited. This is particularly true in the case of molds.

As soon as the press of present urgent problems is somewhat eased, it is contemplated that a survey of the chemical activities of some of these lesser known organisms will be inaugurated. At present no work along this line is being conducted.

RESEARCH PROJECT RRL-5-(7)-F-4. - The Development of Industrial Fermentations Using Corn, Wheat, and Other Cereal Crops or Products Derived From Them as Substrates.

The project represents the core of the work of the Fermentation Division, as it is the new fermentation processes developed in this connection which represent actual increased use of the Laboratory's commodities.



RESEARCH PROJECT RRL-5-(7)-F-4. (Continued)

Two processes are now in the developmental stages, those producing penicillin and itaconic acid. Work on the penicillin problem, conducted for the past three years, has formed the basis for an industry which in 1945 will produce approximately 4,000 billion units of penicillin, enough to treat about 4,000,000 hospital cases. The use of corn steep liquor and lactose in the culture medium, both developed in this Laboratory, will result in the consumption of approximately 12,000,000 pounds of the former and 8,000,000 pounds of the latter during the present calendar year.

During the past six months, the itaconic acid process, using Aspergillus terreus in surface culture, has been given to the public. Although there is still room for much improvement in this process, it can, at present, produce one pound of itaconic acid, of a grade suitable for use in the plastics industry, from each three pounds of glucose used.

The development of other new processes will be undertaken as opportunity offers.

RESEARCH PROJECT RRL-5-(3)-F-5. - The Development of Industrial Fermentation Processes Using Agricultural Residues or Products Derived from Them as Substrates.

This project, which is adequately described in the title, is in abeyance at the present time because of the press of other work and in view of the Synthetic Liquid Motor Fuels Project being carried on by other Divisions of this Laboratory.

RESEARCH PROJECT RRL-5-(3,7)-F-6. - The Isolation, Identification, and Utilization of Chemicals Produced by Fermentation.

In the course of the survey referred to in one of the projects above, new compounds will be encountered. These must be isolated, purified, and identified; or if they are new compounds, their structure will, in many cases, have to be determined. At the present time, work on this project has largely to do with the chemistry of penicillin, although work has recently been completed on the structure of a new acid which is produced in small amounts during the itaconic acid fermentation.

Another phase of the work under this project will be the synthesis of derivatives and the development of new industrial uses for fermentation products.

Agricultural Motor Fuels Division

RESEARCH PROJECT RRL-5-(3,7)-MF-1. - Pilot Scale Technological Investigations of the Production of Agricultural Motor Fuels, and Byproducts by Fermentation.

A fermentation pilot plant complete with cooking, fermenting, distilling, evaporating, and drying equipment capable of processing two



RESEARCH PROJECT RRL-5-(3,7)-MF-1. (Continued)

hundred bushels of grain per day has been designed and installed. This plant has been employed for the production of anhydrous alcohol and butylene glycol from cereal grains. The fermentation of acid-hydrolyzed agricultural residues to ethyl alcohol, acetone, and butyl alcohol for use as fuel adjuncts is now under investigation.

RESEARCH PROJECT RRL-5-(3,7,8)-MF-2. - Formulation, Development, Investigation, and Testing of Gaseous, Liquid and Solid Motor Fuels from Agricultural Materials.

In order to investigate the suitability of liquid motor fuels derivable from agricultural raw materials, two standard single-cylinder test engines and a six cylinder Plymouth engine have been installed. Accessory equipment is also available for testing the effect on power output of inlet air temperature and pressure, compression ratios, cylinder and manifold injection, spark advance, and engine speed. Extensive data have been compiled on the behavior of various agricultural motor fuels with special reference to the octane number of various blends. The water tolerances of alcohol-hydrocarbon mixtures have been measured and the antiknock properties of ironpentacarbonyl in alcohol fuels have been demonstrated. A producer gas generator employing corn cobs has been designed.

RESEARCH PROJECT RRL-5-(3,7,8)-MF-3. - Laboratory-Scale Technological Investigations of the Production of Motor Fuels and Byproducts from Agricultural Materials.

Investigations are being conducted to evaluate fermentation methods for the production of motor fuels from agricultural materials. These studies have involved a survey of sources of fermentable carbohydrates as well as the most practical methods of cooking, saccharifying, fermenting, and recovering byproducts from each material.

An improved procedure has been developed for cooking cereal grains which increases the rate and degree of saccharification when malt is added. Conditions have been established for the production of industrial alcohol from cereal grains substituting acid for malt enzymes. The fortification of fermentation byproduct feeds with vitamins is being studied by cultivating various microorganisms in the fermenting residues. In this connection, an organism has been found which is capable of synthesizing, under controlled conditions, unusually large quantities of vitamin B<sub>2</sub>.

Oil and Protein Division

RESEARCH PROJECT RRL-5-(7,8)-OE-1. - Composition and Processing of Soybeans and Other Oilseeds, and of Corn and Other Cereal Grains for Oils and Proteins.

Research in this project has been confined mainly to soybeans. Although a crop which lends itself well to large-scale production, the present methods of industrial processing do not give products of superior quality.



RESEARCH PROJECT RRL-5-(7, 8)-OP-1. (Continued)

Consequently, considerable effort has been expended to obtain new methods of separating the oil and protein from the other constituents. It has been determined that alcoholic extraction of soybeans gives oil and protein which are superior in color and taste to expeller or hexane-extracted products. Research on a process of extraction with alcohol, on the flavors which develop in soybean oil and meal, and on the minor constituents which may be responsible for flavors is being continued.

RESEARCH PROJECT RRL-5-(8)-OP-2. - Modification of Linseed, Soybean, and Other Vegetable Oils of the Northern Region and the Components Thereof.

Research in this project has been restricted primarily to investigations on the shifting of double bonds in polyunsaturated fat acids. Isomeric products obtained by alkali isomerization of linoleic acid have been isolated and identified. Similar products from linolenic acid have been investigated, but isolation of pure products has proved to be very difficult. Catalytic methods of isomerization which do not cleave the glyceride molecule are being investigated, and potential uses of the resulting conjugated oils are being evaluated.

RESEARCH PROJECT RRL-5-(8)-OP-3. - Polymerization Reactions of Soybean and Linseed Oils, Their Fat Acid Components and Derivatives.

Soybean and linseed oils contain appreciable amounts of polyunsaturated fat acids which are potential sources of industrially important new materials. By heating these acids, polymeric (dimeric and trimeric) fat acids are obtained which can be used to make a wide variety of polymeric derivatives. The ethylene glycol polyester of these polymeric fat acids can be made into a rubber replacement for some specific uses, and the ethylene diamine polyamide is being produced on a semi-commercial scale and is being used for heatsealing of food containers. Many other interesting polymers have been investigated, but their utility has not been established. Research on other modifications of the polyunsaturated fat acids are being investigated to give new derivatives which may have industrial importance.

RESEARCH PROJECT RRL-5-(7,8)-OP-4. - Soybean, Linseed, Corn, and Other Protein Investigations.

Two major uses for industrial proteins are synthetic fibers and plywood adhesives. A process has been developed for using Merco corn gluten or a special grade of soybean meal as an extender for phenolic-resin, plywood glue. Plywood prepared with glue having a ratio of two parts of proteinaceous material to three parts of resin, meets specifications for exterior plywood. Zein fibers have been prepared by the wet-spinning method. Dry tensile strengths equal to the good grades of wool and wet strengths equal to about two-thirds that of wool have been obtained. Additional research is now under way.



Agricultural Residues Division

RESEARCH PROJECT RRL-5-(3)-R-1. - Development of Papers and Pulps from Agricultural Residues.

Alpha-cellulose produced from rice and barley straws because of the narrow diameter of the fibers has been found particularly suitable for use in the manufacture of air filters in gas masks.

The development of methods for pulping soybean straw, sugarcane bagasse, hemp hurds, and other cereal straws for the production of corrugating board used in shipping containers has been of material assistance to the 28 mills operating in the strawboard industry in supplementing the shortage of wheat straw formerly used solely as the raw material.

Studies are being continued to discover those properties of cellulose pulps produced from agricultural residues which adapt them for use in specialty papers.

RESEARCH PROJECT RRL-5-(3)-R-2. - Development of Building Materials and Miscellaneous Products from Agricultural Residues.

A pilot plant study to develop an economical process for manufacturing structural insulating building board on a scale small enough for a rural industry has demonstrated the technical, business, and cost problems which must be solved. This study is almost completed. Work is now under way to develop better methods for the use of agricultural residues in the manufacture of such building materials as structural insulation and hard board.

Noreseal, recognized as a satisfactory cork substitute for use as a liner in crown seals for bottling beverages and foods, is composed of about 30 percent peanut shell pith, together with glue, glycerine, glucose, and formaldehyde. Pilot plant studies to develop manufacturing details and cost data are almost complete.

As a result of cooperative work with the Bureau of Aeronautics, ground corncobs have been adopted by the Navy as the standard soft grit for the air-blast removal of carbon and oil deposits from airplane engines.

RESEARCH PROJECT RRL-5-(3)-R-3. - Development of Plastics and Plastic-like Materials from Agricultural Residues.

Contrary to past belief it has been demonstrated that wood flour, long used as the filler component of phenol-formaldehyde plastics, can be satisfactorily replaced with dry ground agricultural residues, such as corncobs, flax shives, hemp hurds, and straw. The phenol-formaldehyde plastics were developed around the use of wood flour as filler, and consequently the resin component selection is most favorable to wood flour usage. When a proper selection of the resin component is made with agricultural residue flours, the plastics produced are not only equal to wood flour-filled plastics in physical properties generally, but in some cases, such as with the use of corncob flour, have proved superior to wood flour-filled plastics.



RESEARCH PROJECT RRL-5-(3)-R-3. (Continued)

General-purpose phenol-formaldehyde molding powders using wood flour as filler contain about 47 percent phenol-formaldehyde resin. Plastics of equal physical properties are now made possible by the use of agricultural residue flours, a newly discovered plasticizer, and only 25 percent of phenol-formaldehyde resin.

The continuous process for the saccharification of agricultural residues represents a new potential source of large amounts of lignin at low costs. A study is being made of the possibility of using this lignin to produce thermoplastic and thermosetting molding compounds.

RESEARCH PROJECT RRL-5-(3)-R-4. - Investigation of Chemical Constitution, Reactions, and Physical Properties of Lignin Obtained from Agricultural Residues.

Due to more pressing war problems, these studies have been held in abeyance until recently. Work is now underway on the chemical reactions and the possibilities of producing derivatives of lignin resulting from the saccharification of agricultural residues. Work is directed particularly towards converting the lignin to liquid fuel products and to compounds useful in producing plastics.

RESEARCH PROJECT RRL-5-(3)-R-5. - Development of Industrial Uses for Hemicelluloses and Derivatives from Agricultural Residues.

Methods of producing the following products from furfural have been developed: 1,3-and 1,4-pentadiene and 2-vinyl furan for use in elastomers and resins; tetrahydrofuran and dihydropyran for possible use as liquid fuels; and certain important pharmaceutical intermediates. This work is continuing.

A process for converting 2,3-butylene glycol to 1,3-butadiene by pyrolysis of the glycol diacetate at 590°C. has been developed through the laboratory and a 100 lb./day pilot-plant stage. The process, after being operated in a one-ton per day pilot plant by an industrial company, was reported to the Office of Rubber Director by a consulting engineering firm as being entirely practical. Engineering, plant construction, and cost details constitute a part of the report. The butadiene yield is 88 percent of theory, together with the production of about 10 percent of methyl ethyl ketone, while acetic acid losses do not exceed one percent.

A continuous process for saccharifying agricultural residues has been developed which will produce 2,000 pounds of glucose, 1,600 pounds of xylose, 200 pounds of furfural, and 1,000 pounds of lignin from 3.3 tons of corncobs, bagasse, peanut shells, or other farm wastes. By the use of suitable micro-organisms the sugars, glucose and xylose, can be fermented to a number of liquid fuels. The xylose can be converted to furfural in high yield; which may be used directly or in the form of certain of its derivatives as liquid fuel. Lignin is converted by hydrogenation to liquids of possible fuel value.



Starch and Dextrose Division

RESEARCH PROJECT RRL-5-(7)-SD-1. - Starch Granule Formation, Properties, and Composition.

Most of the industrial uses for starch are dependent on its colloidal and physical properties in water. Work has therefore been conducted on evaluating the factors which affect the properties of the pastes, in particular, the effect of the degree of granule breakage as brought about by rate of stirring and pasting conditions on viscosity. The effect of commercial processing conditions on the properties of starch has been studied. The function of sulfur dioxide which is used by the corn wet-milling industry to soften corn prior to milling has been found to be due to the disintegrating action of sulfur dioxide on the protein matrix in which the starch is imbedded in the endosperm. Starch sponges, prepared by freezing starch pastes, have been tested as surgical dressings and incorporated in a variety of processed foods to improve their palatability and characteristics. New uses for starch sponge are promising.

RESEARCH PROJECT RRL-5-(7)-SD-2. - Starch Structure, Properties, and Hydrolysis.

Practically no starch is being used by industry for the production of chemical raw materials. Use of starch for such purposes is dependent on the development of an economical method of fractionating starch and on the characteristics of the components and their derivatives. A wide variety of organic compounds has been tested as possible fractionating agents. It has been found that many different classes of organic compounds can be used as fractionating agents, some being commercially available and cheap. Work on fractionating starch is being translated to a pilot-plant scale. A method for determining the presence and number of branching "isomaltose" units in starch has been developed and is being applied to characterize the amylopectin component and fractions of starch. Retrogradation of starch, that is, its spontaneous precipitation from starch-water sols or pastes, is being thoroughly investigated from the standpoint of rate and composition of retrograded product. This information is useful in extending the applications of corn starch in the textile and sizing industry.

RESEARCH PROJECT RRL-5-(7)-SD-3. - Starch: Chemical Conversion.

The properties of such starch derivatives as whole starch acetate and methyl starch are unsuitable for industrial use. These products are too brittle for use as plastics or fibers. The amylose component of starch, however, has been found to have excellent film-, fiber-, and plastic-forming properties. Various derivatives of amylose, such as amylose acetates, propionates, and butyrates, as well as methyl and ethyl amylose, have been prepared and their film and fiber properties are being compared with the analogous whole starch and amylopectin derivatives. Optimum conditions for converting starch by high-temperature distillation to levoglucosan have been determined. The cost of producing levoglucosan on an industrial scale is being ascertained.



RESEARCH PROJECT RRL-5-(7)-SD-4. - Dextrose: Modification,  
Condensation, and Polymerization.

Because of its cheapness and availability, dextrose is a potential raw material for large-volume industrial uses. Major emphasis at the Northern Regional Research Laboratory has been placed on the hydrogenolysis of dextrose and its derivatives and on the nitric acid oxidation of dextrose to saccharic acid. Previous work, which showed that a complex mixture of alcohol, glycols, glycerol, and tetrahydroxy compounds are formed on hydrogenation has been confirmed. The conditions or catalyst which will preferably form a major product have not yet been discovered. The method for producing saccharic acid has been improved and the raw-material costs determined. The process is being translated to a pilot-plant scale. Uses for saccharic acid or its derivatives are under investigation.

RESEARCH PROJECT RRL-5-(7)-SD-5. - Dextrose: Rearrangement and Degradation.

This phase of dextrose chemistry was temporarily dropped two years ago in order to investigate cereals more available than corn as possible sources for starch and glucose. Two new processes, the Alkali Process and the Batter Process, for producing wheat starch from wheat flour were developed. The principles of the Batter Process have been adapted to an industrial scale by a number of companies. One of these operates the process in a converted beet sugar refinery and is producing about 10 million pounds of glucose sirup annually. The byproduct wheat gluten is converted to sodium glutamate, the entire production of which is being used by the armed forces as a meat flavoring agent in soups.